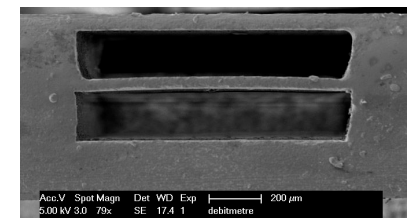
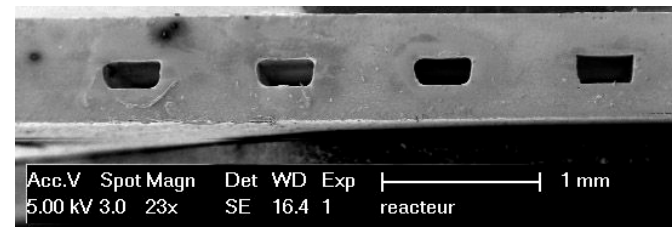
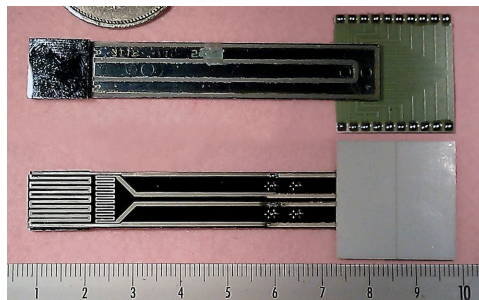
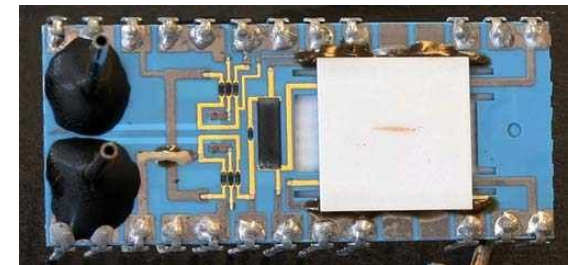
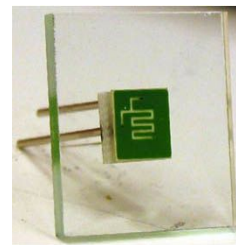
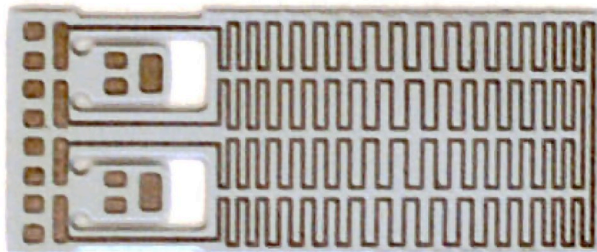


# 3D structuration of LTCC and related technologies for thermal management and microfluidics

Thomas Maeder<sup>1</sup>, Conor Slater<sup>1</sup>, Bo Jiang<sup>1,2</sup>, Fabrizio Vecchio<sup>1</sup>, Caroline Jacq<sup>1</sup>, and Peter Ryser<sup>1</sup>

EPFL-LPM<sup>1</sup> & EPFL-LC<sup>2</sup>



- 1. Introduction**
- 2. Processing for 3D**
- 3. Materials formulation**
- 4. Conclusion & outlook**

# Outline (1)

---

## 1. Introduction

- **Our lab**
- **Thick-film & LTCC**

## 2. Processing for 3D

## 3. Materials formulation

## 4. Conclusion & outlook



## EPFL École Polytechnique Fédérale de Lausanne

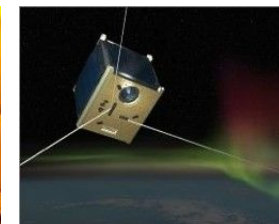
One of the two polytechnical  
schools in Switzerland

10'000	Population on the campus
6'000	Students
3'500	Collaborators
1'400	PhD. Students
250	Professors
550	MSFr budget / year
70	Companies on site
10	New start-ups / year

- **SB:** School of basic sciences
  - Chemistry, mathematics and physics
- **SV:** School of life sciences
  - Genomics, neurosciences, ...
- **ENAC:** School of civil + environmental engineering & architecture
  - Architecture, civil engineering, environmental sciences and technologies
- **I&C:** School of informatics and communications
  - Fundamental & applied computer science, communication systems
- **STI: School of engineering**
  - Electricity, materials, mechanical engineering, biomedical engineering, **microtechnology**

- **IMX:** Institute of materials science & engineering
  - Metals, ceramic, polymers, composites, powders, bio-/nanomaterials,...
- **IEL:** Institute of electrical engineering
  - Circuits & devices, power & energy, telecom, computers...
- **IGM:** Institute of mechanical engineering
  - Solid + fluid mechanics, heat + mass transfer, biomechanics, control...
- **IBI:** Institute of bioengineering
  - Biology, chemistry, biomaterials, ... (*with school of life sciences*)
- **IMT: Institute of microengineering**
  - Microsystems, robotics, optics, production, ...
  - Inherently multidisciplinary
- *+ several technology centres:*

<http://imt.epfl.ch>





# IMT - Locations



***The new IMT: 1 Institute on 2 Sites***

***Lausanne + Neuchâtel  
Campus***



***Distance: ~70 km or 45 min***



***Close  
collaborations***

**csem**

**Hes·SO**  
Haute École Spécialisée  
de Suisse occidentale

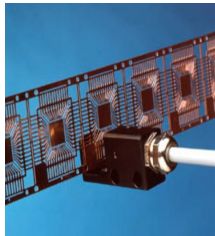
***Industry***

<http://imt.epfl.ch>

## *Laboratoire de Production Microtechnique*

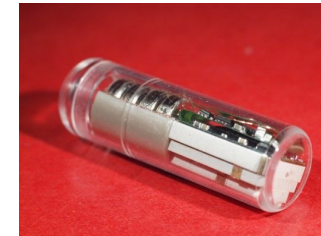
### **Vision & $\mu$ -Assembly**

Director : Prof. J. Jacot  
Group head : A. Dufaux



### **Product design**

Director : Prof. P. Ryser  
Group head : E. Meurville



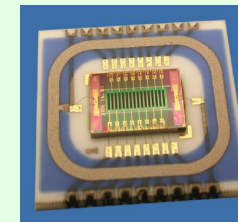
### **Production strategy**

Director : Prof. M.-O. Hongler



### **Thick-film technology**

Director : Prof. P. Ryser  
Group head : Th. Maeder





# Topics of LPM thick-film group @ EPFL

## Harsh Environments

Aerospace - Implantable systems  
Chemistry – Nuclear - Reactive materials  
High-temperature processes

## Load sensors

Force / pressure sensing  
Integration in packages  
Structuration  
Medecine / rehabilitation

## Technologies

### LTCC Thick-film

## Fundamentals

Materials science  
Processing  
Theory + modelling

## Ceramic microfluidics

Microreactors / calorimeters  
Gas sensors  
Fuel cells

## Advanced Packaging

MEMS – Integrated functions  
Hermetic – temperature control  
Integrated sensors/actuators/fluidics  
Structuration – sacrificial layers

# Thick-film technology - introduction

- **Thick-film circuit : series of layers**

- Screen-printing of layers with a mask
- Direct dispensing (prototypes)

- **Each layer comes as a paste:**

- Functional material (as powder)
- Organic vehicle: binder + solvent

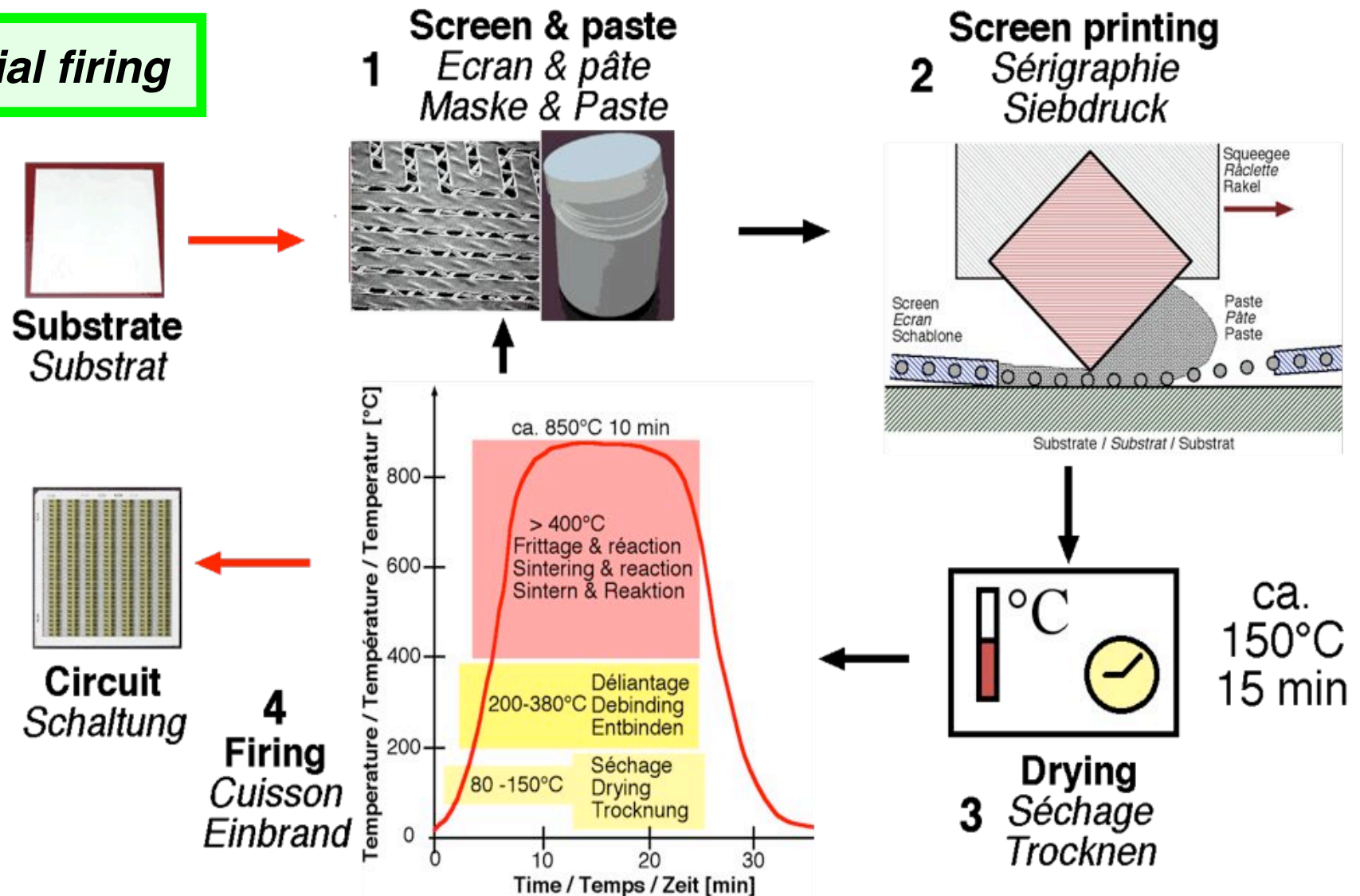
- **Materials**

- Conductors
- Resistors
- Dielectrics
- ...**and more!**



# Thick-film - process flow

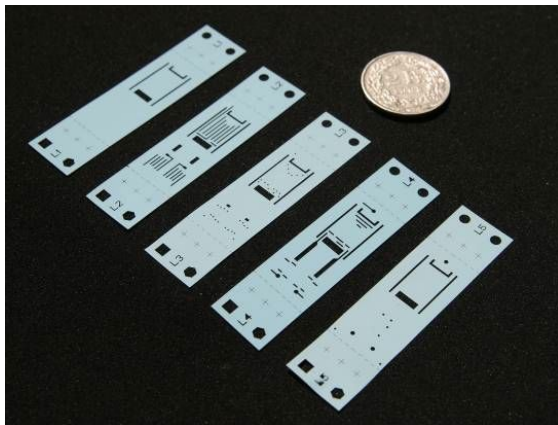
## Sequential firing



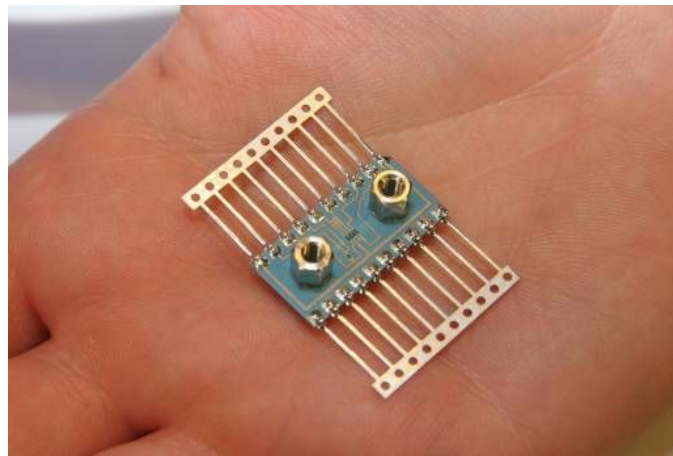
# LTCC - introduction - the material?

## Low-Temperature, Cofired Ceramic

- Sheets of sintered ceramics (blue, white or black)
- You're carrying it unawares (mobile phone, car ignition)
- Relatively new material (<20 yrs)
- Developed for highly integrated electronics

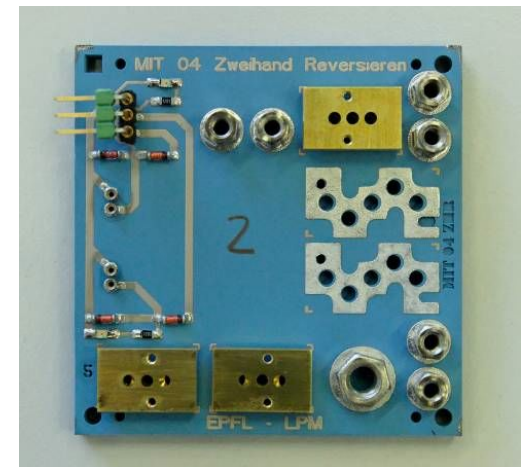


*raw sheets of LTCC  
(micro-reactor)*



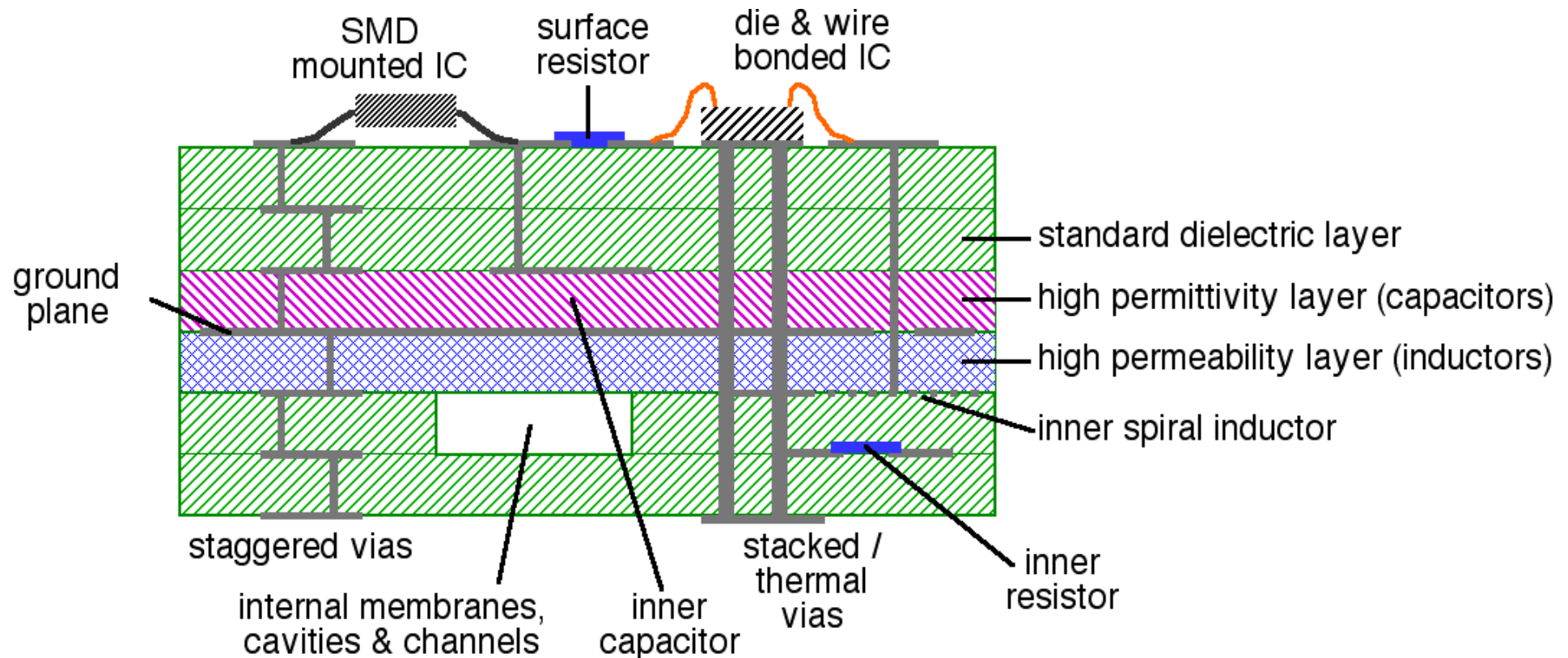
*micro-flow sensor  
assembled*

*fluidic circuit,  
management of valves  
with SMD electronics*





# 3-D structuration of LTCC





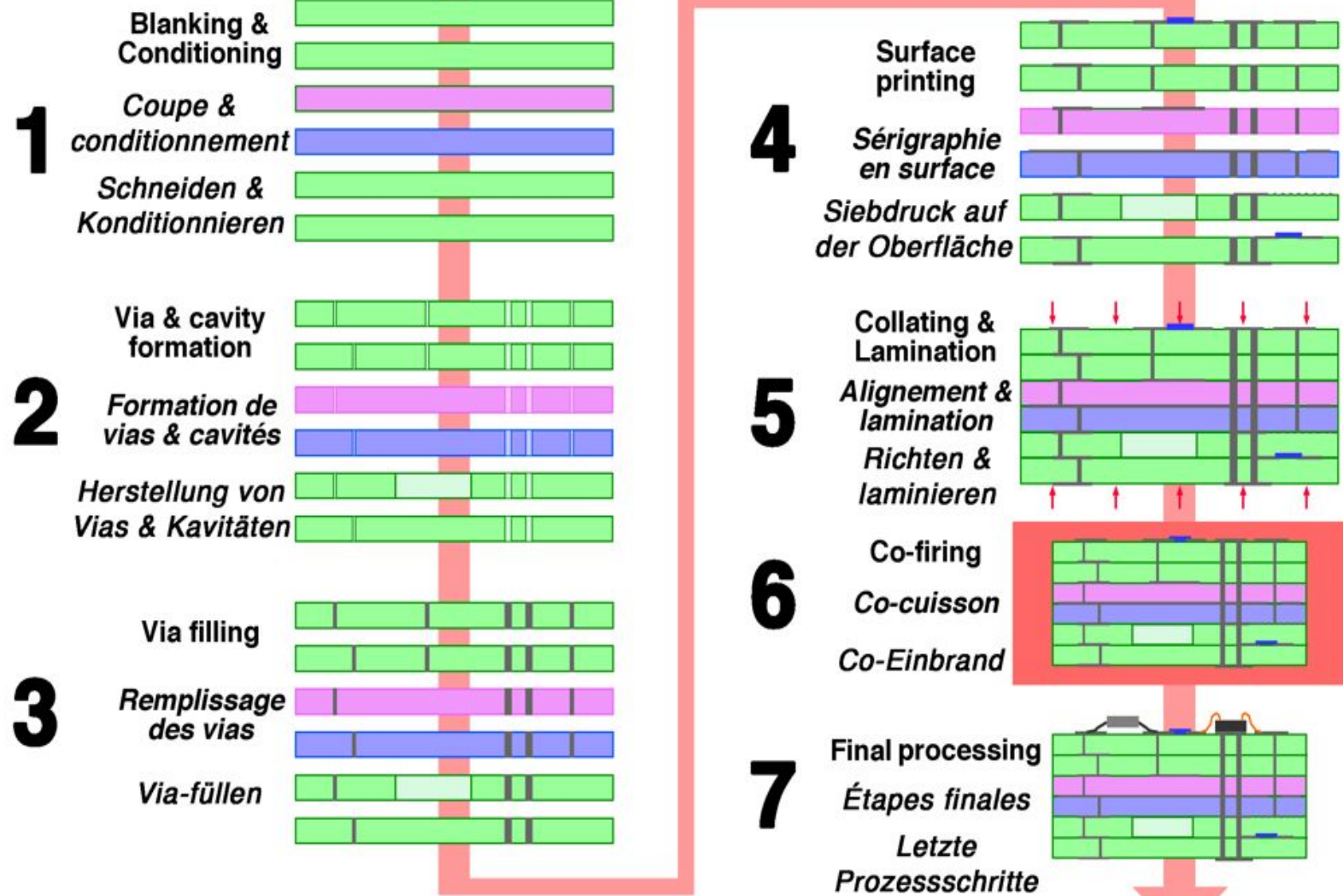
# LTCC vs. alumina for sensors

Material	LTCC (DP 951)	Al <sub>2</sub> O <sub>3</sub> (96%)	Ratio
Minimal thickness [mm]	0.04	0.17	0.24
Short-term strength [MPa]	320	600	0.53
10 year strength [MPa]	110	270	0.41
Young's modulus [GPa]	110	320	0.34
Thermal conductivity [W/m]	3	25	0.12
Design strain [ppm]	1'000	800	1.25
Flexural sensitivity [kN <sup>-1</sup> ]	5.68	0.11	53
Thermal resistance [K/W]	8'333	235	35

- LTCC more sensitive
- Good 3D structurability
- Compatibility with standard mounting (SMD)

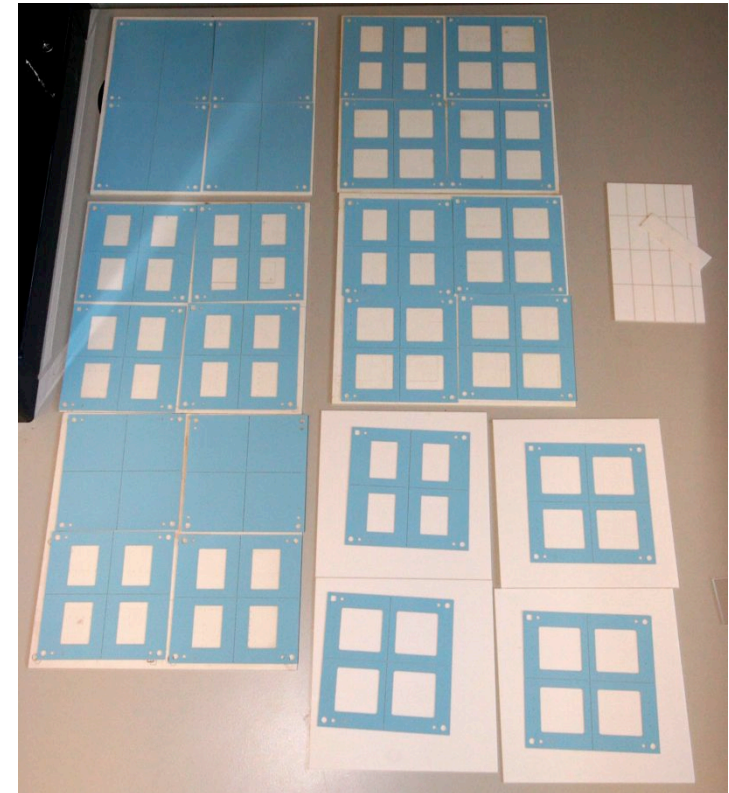
# LTCC - process flow

## Co-firing



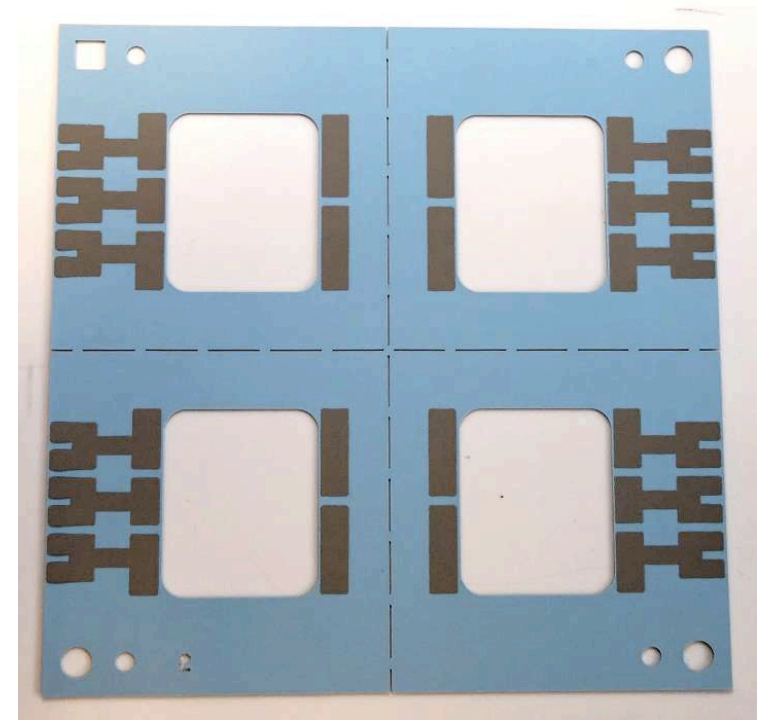
# LTCC - principle - 1 & 2

1. Raw sheets easily **cut**  
(laser, punch tool)
2. Formation of vias & cavities
3. Vias filled for interlayer contacts
4. Layers individually printed  
(multilayer circuits )
5. Stacking & lamination of layers to  
get  
a 3D structure
6. Firing  
-> sintering, monolithic circuit
7. Individualisation and post-firing  
(assembly by soldering)



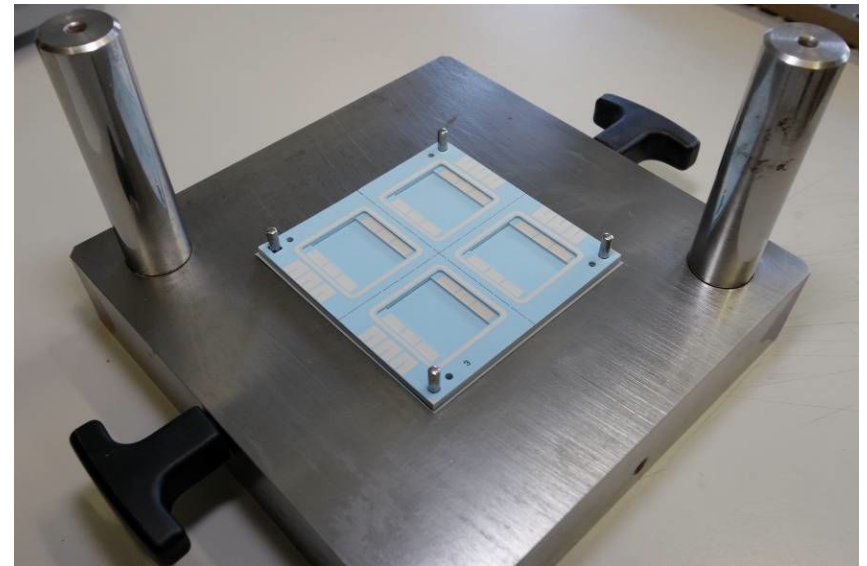
# LTCC - principle - 3 & 4

1. Raw sheets easily cut  
(laser, punch tool)
2. Formation of vias & cavities
3. Vias filled for interlayer contacts
4. Layers individually printed  
(multilayer circuits )
5. Stacking & lamination of layers to  
get  
a 3D structure
6. Firing  
-> sintering, monolithic circuit
7. Individualisation and post-firing  
(assembly by soldering)



# LTCC - principle - 5

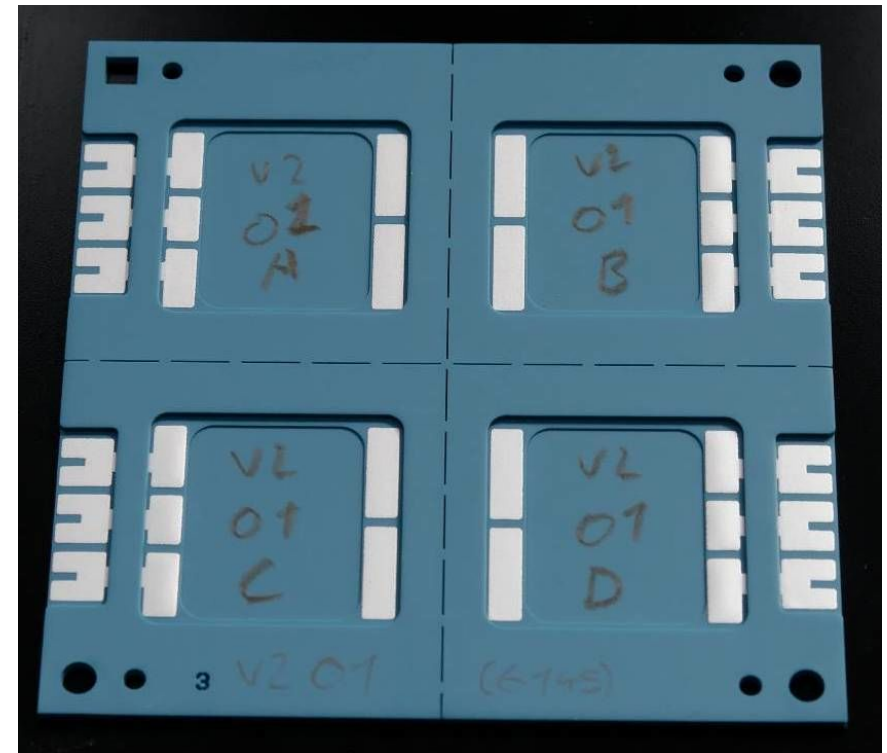
1. Raw sheets easily cut  
(laser, punch tool)
2. Formation of vias & cavities
3. Vias filled for interlayer contacts
4. Layers individually printed  
(multilayer circuits )
5. **Stacking & lamination of layers to  
get  
a 3D structure**
6. Firing  
-> sintering, monolithic circuit
7. Individualisation and post-firing  
(assembly by soldering)





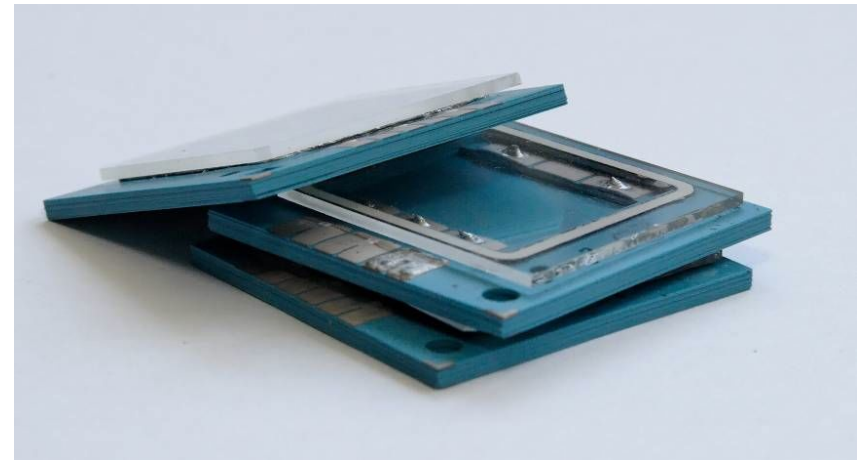
# LTCC - principle - 6

1. Raw sheets easily cut  
(laser, punch tool)
2. Formation of vias & cavities
3. Vias filled for interlayer contacts
4. Layers individually printed  
(multilayer circuits )
5. Stacking & lamination of layers to  
get  
a 3D structure
6. Firing  
-> sintering, monolithic circuit
7. Individualisation and post-firing  
(assembly by soldering)



# LTCC - principle - 7

1. Raw sheets easily cut  
(laser, punch tool)
2. Formation of vias & cavities
3. Vias filled for interlayer contacts
4. Layers individually printed  
(multilayer circuits )
5. Stacking & lamination of layers to  
get  
a 3D structure
6. Firing  
-> sintering, monolithic circuit
7. Individualisation and post-firing  
(assembly by soldering)



## 1. Introduction

## 2. Processing for 3D

## 3. Materials formulation

## 4. Conclusion & outlook

- **Basic processing for 3D structures**
- **Handling issues**
- **Sacrificial volume materials (SVMs)**
- **Lamination & firing**

# LTCC structuration methods

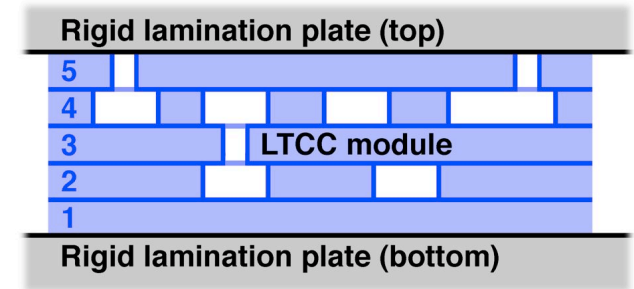
## Features

- Vias
- Channels
- Membranes
- Beams

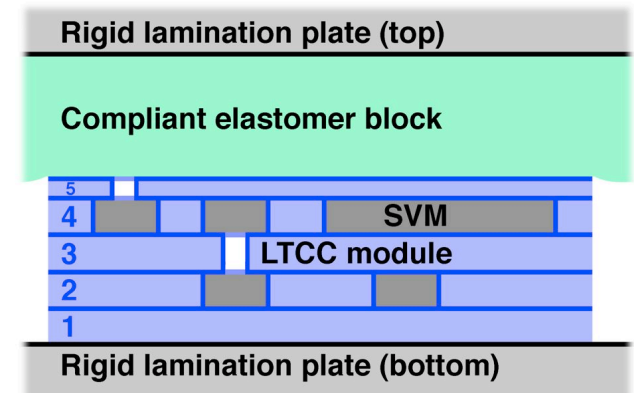
## Three lamination routes

- 1) Cutting & stacking, flat
- 2) Cutting & stacking, +SVM
- 3) Structuration by SVM

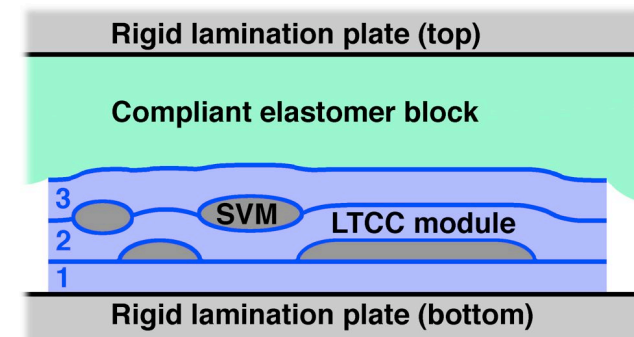
1



2



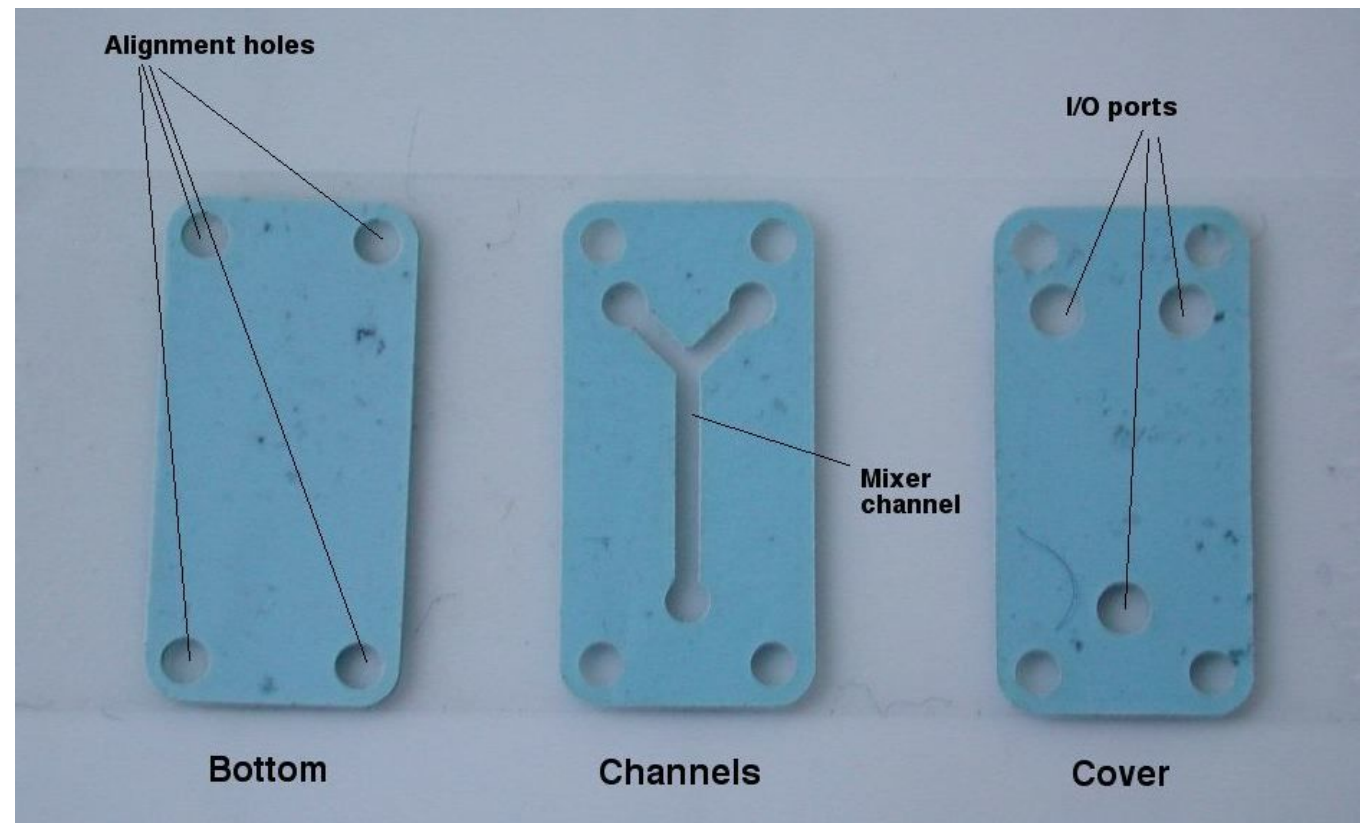
3



# Cutting & stacking LTCC

## Process

1. Cutting
2. Stacking & laminating
3. Firing

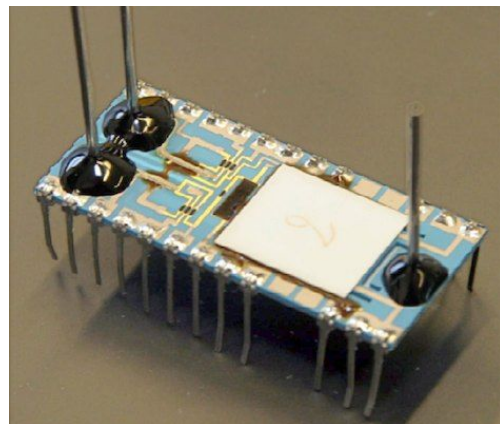
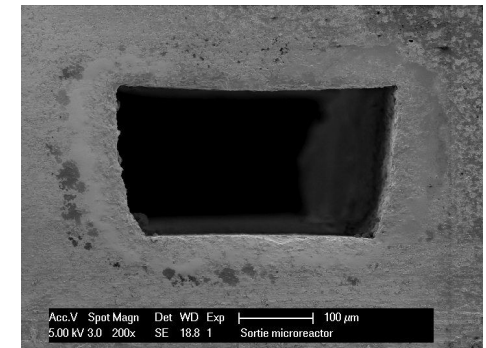
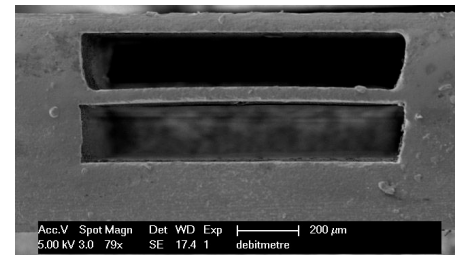
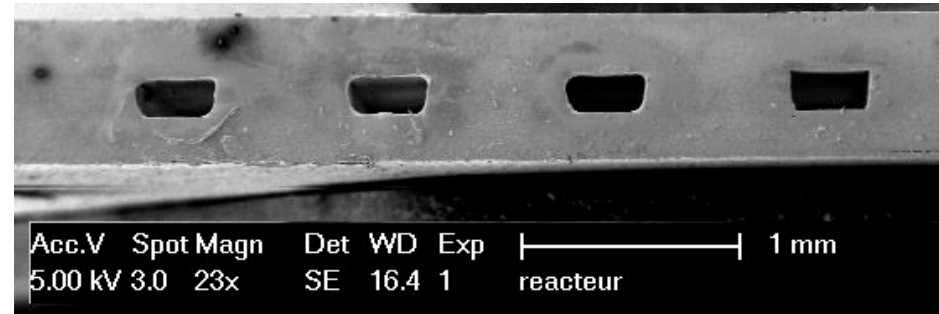


## A simple LTCC micromixer



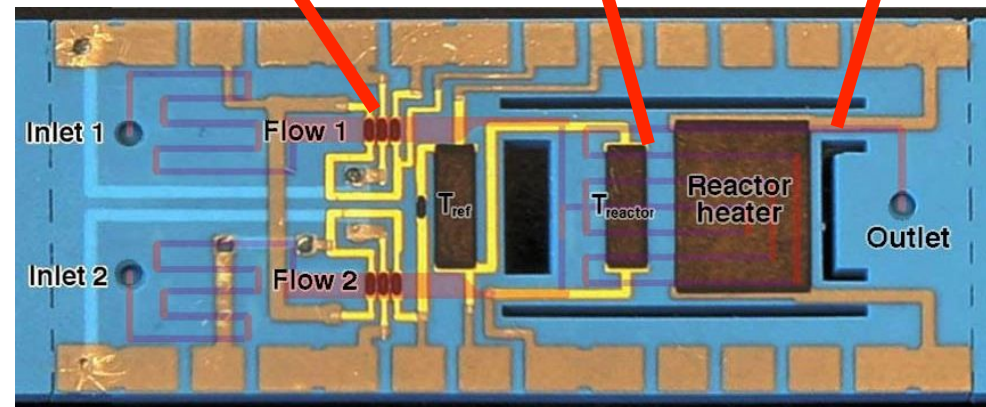
# Ceramic structuration: microreactor

- Complex fluidic circuit (2 layers + membranes)
- Flow sensors
- Heaters
- Channels in support posts
- ...



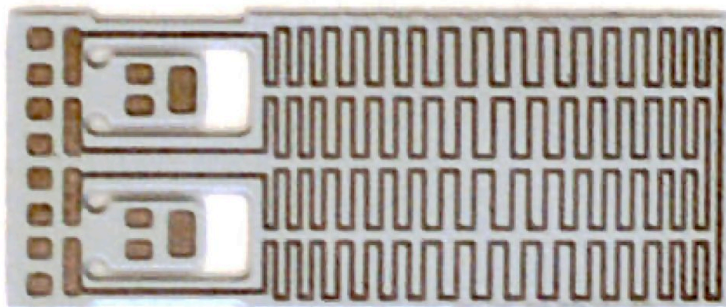
LTCC module +  
fluidic circuit

Complete  
device



# LTCC $\mu$ -SOFC test platform

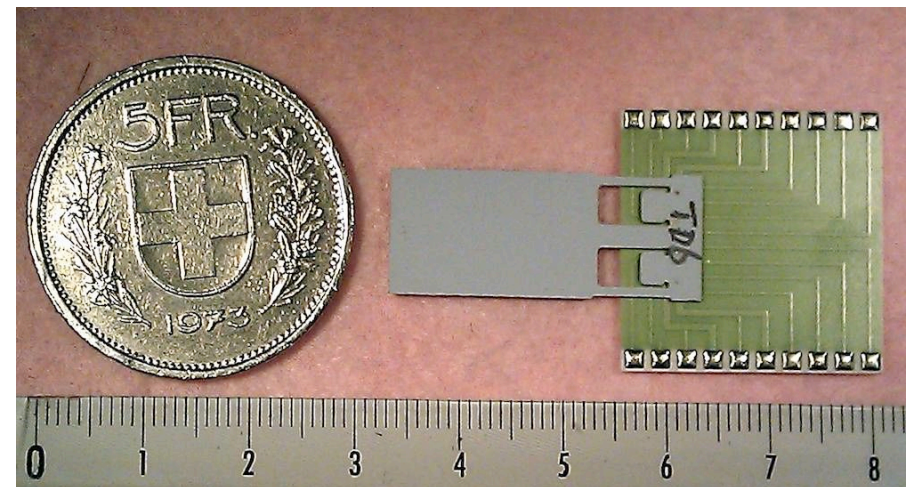
- Thermal decoupling by slender bridges
- Electrical (bottom) & fluidic (top) connections
- Heating & temperature control by Pt meanders
- SMD-soldered to thick-film base for easy connections & cooling of "cold" side



LTCC  $\mu$ -SOFC test platform



Pt track



Platform mounted onto base

# Fluidic multisensor

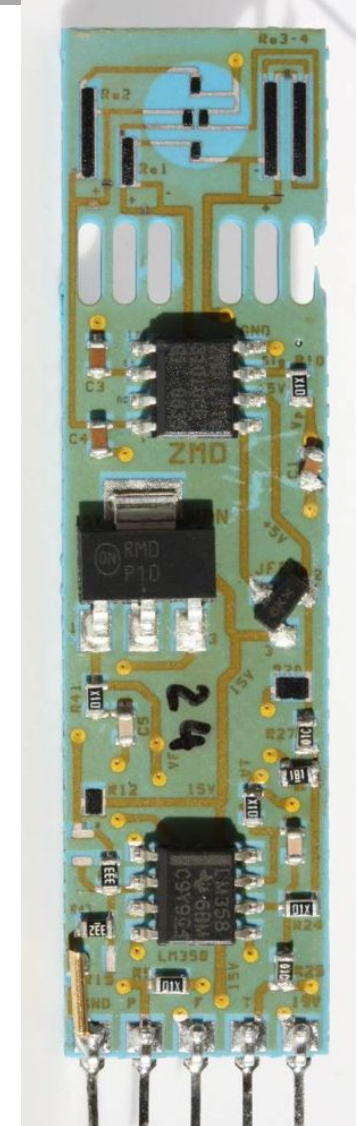
## Application: pneumatic industry (diagnostics)

- **air pressure** 0...6 bar
- **air flow** 0...100 NL/min
- **air temperature** 0...100 °C

## Integration in LTCC

- **3-D multiphysics** sensing
- ...with **integrated electronics**
- ...**SMD** mountable by soldering

First presented at  
Eurosensors 2009,  
Lausanne (CH)

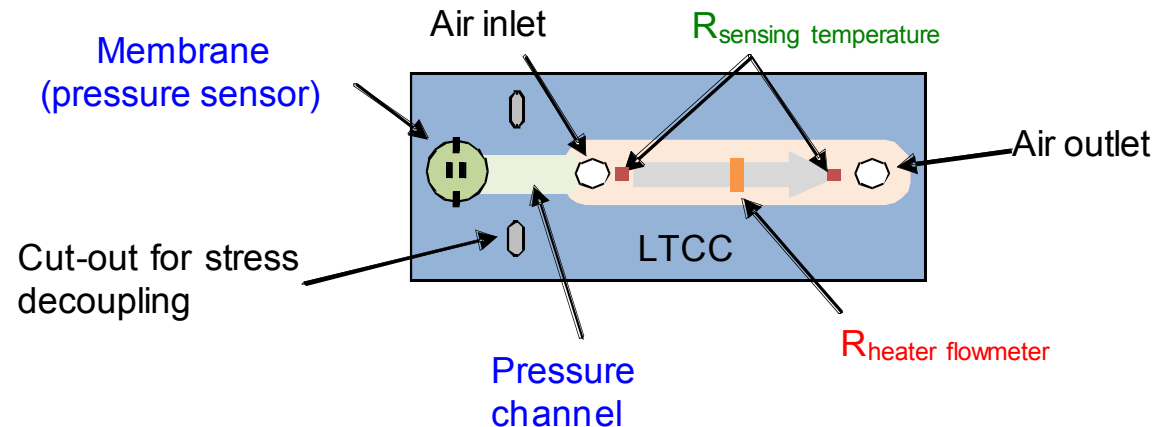




# Multisensor concept for P, T, flow

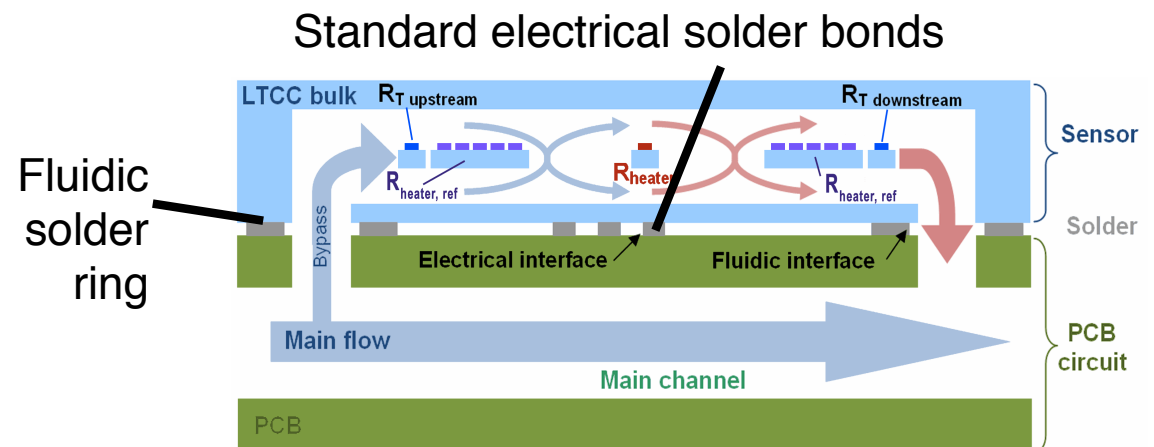
## Integrated LTCC sensor with electronics:

- Pressure
- Flow
- Temperature



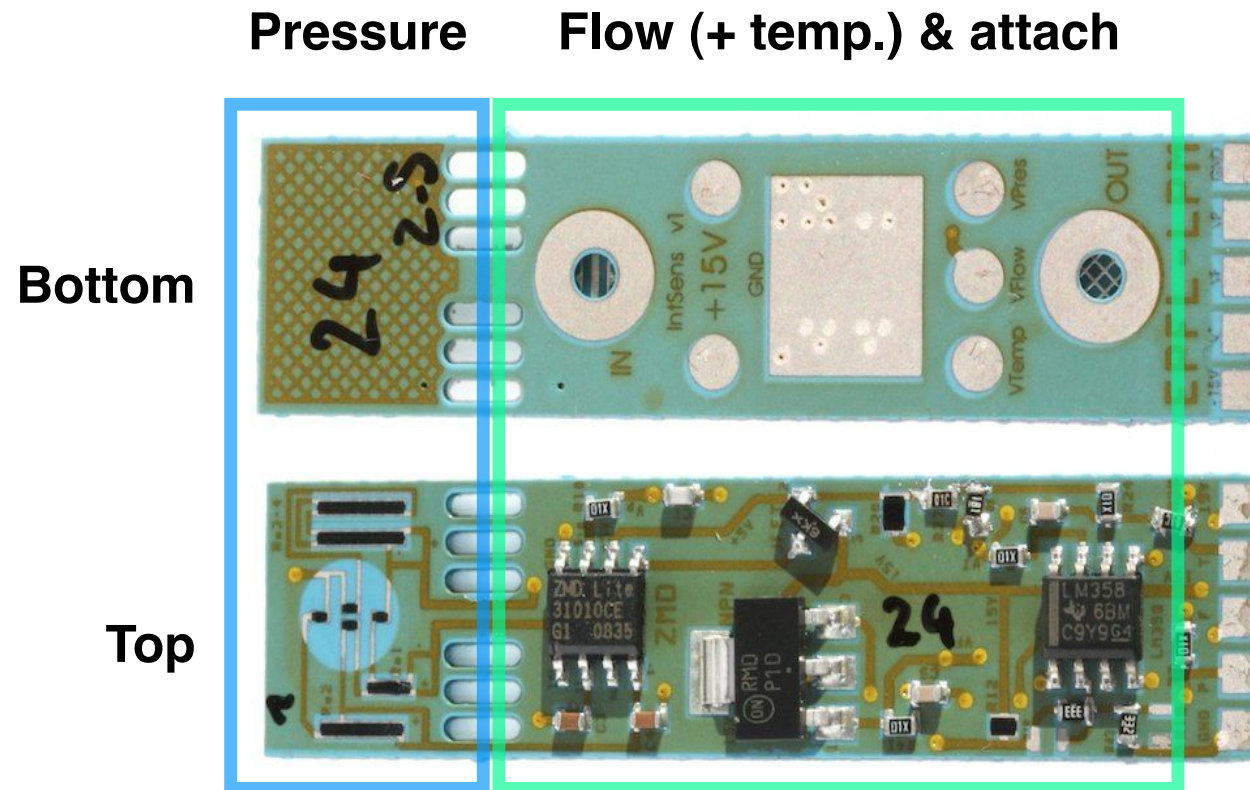
## Sensor properties

- Safe & reliable
- Low-cost
- Easy to integrate - "total SMD" fluidics + electronics



# Multisensor overall layout

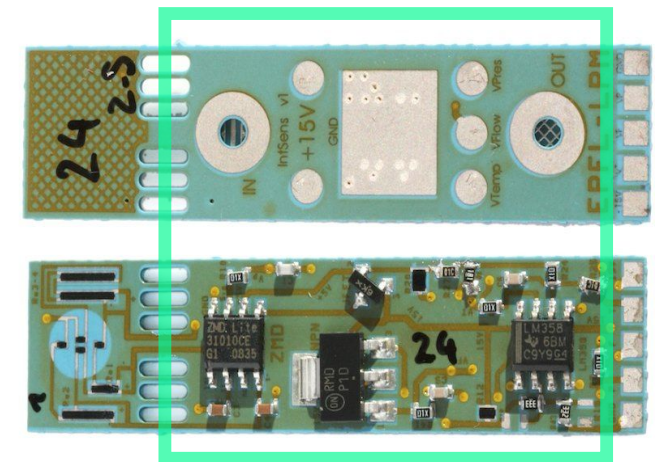
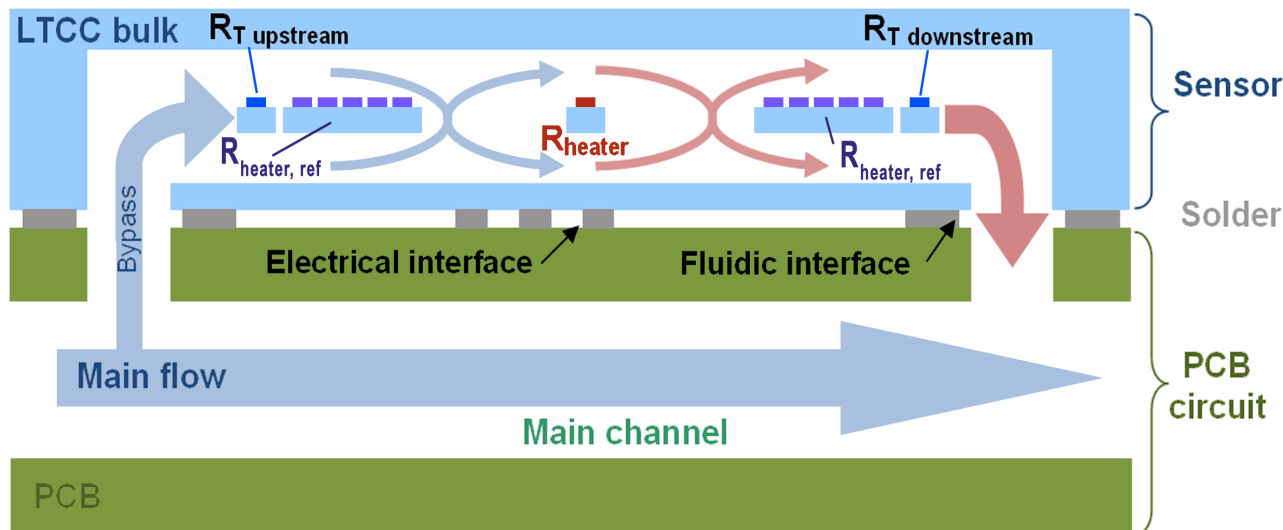
- Long geometry
- SMD pads on flow sensing part ( $\approx$ insensitive to stresses)
- Stress-sensitive pressure sensing part decoupled



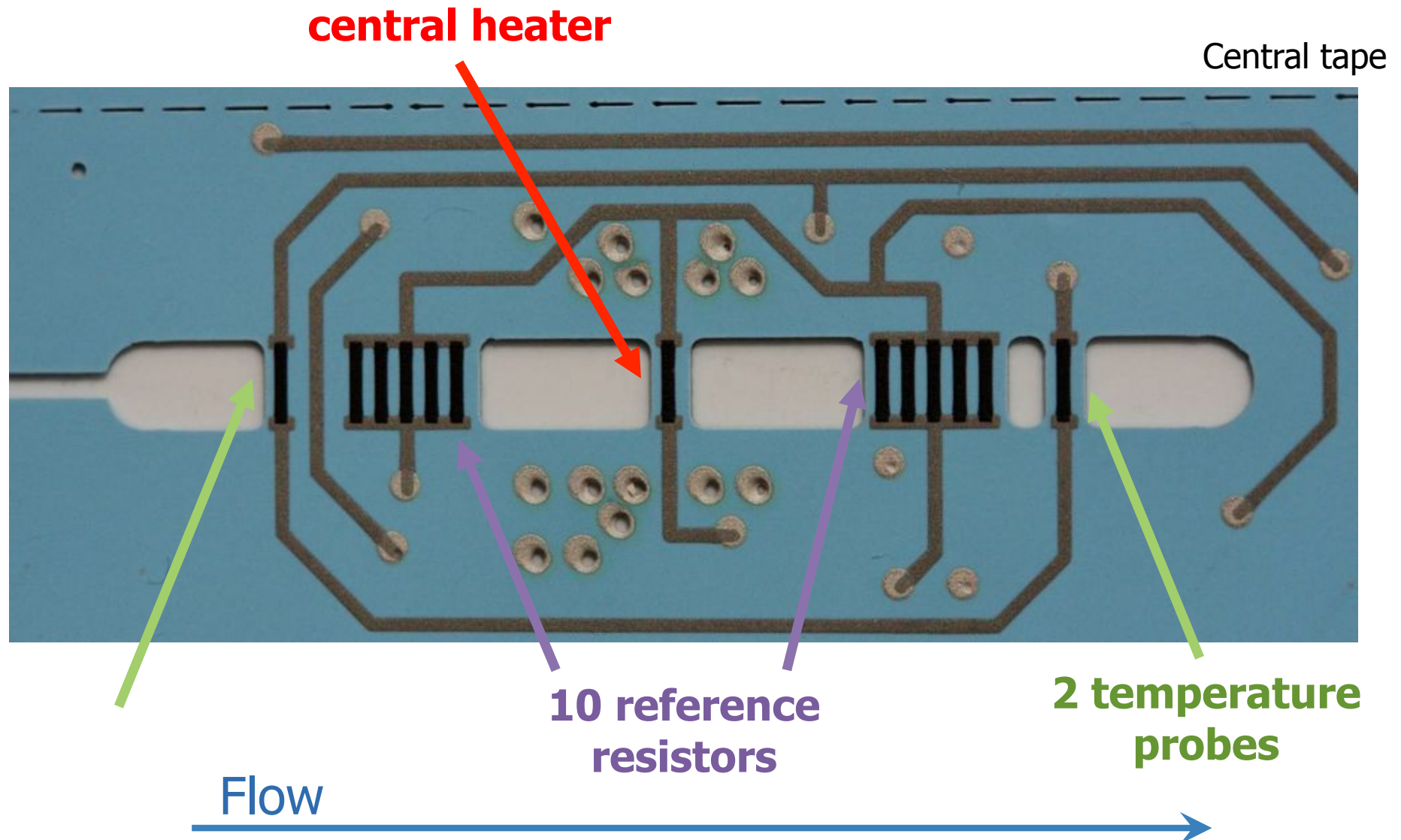


# Multisensor - flow-sensing part

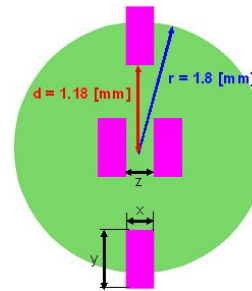
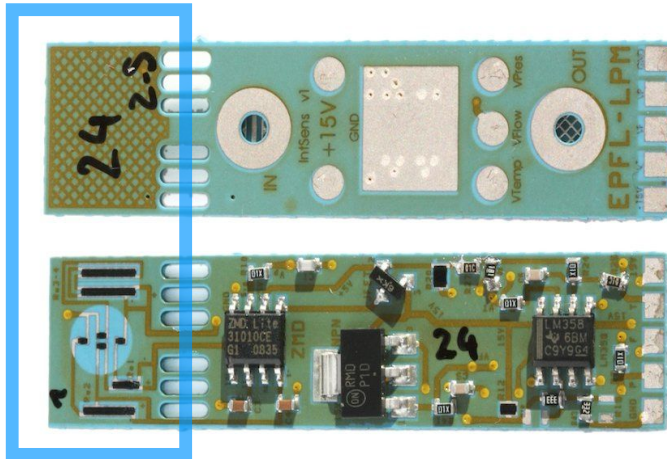
- Measuring + reference thermistors on suspended bridges
- Regulated temperature increment  $\Delta T$ : measuring vs. reference
- High flows: possible bypass in electrical + fluidic PCB
- Extra passive thermistors to measure temperature



# Multisensor - flow & temp. resistor layout

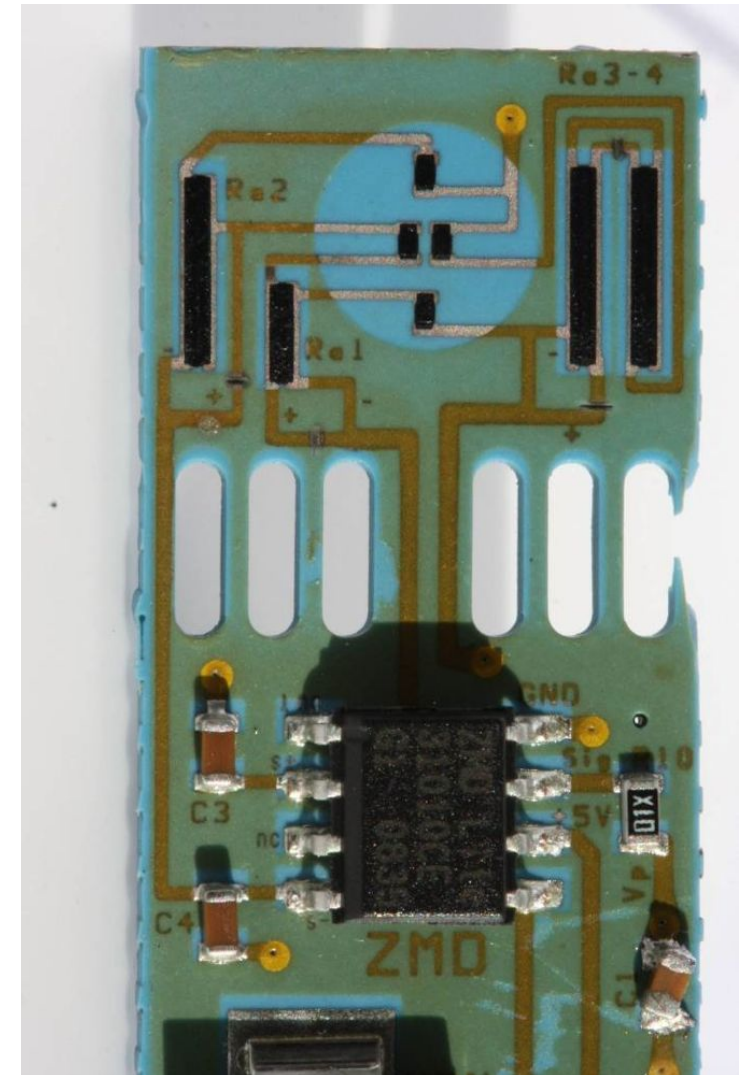


# Multisensor structure - pressure



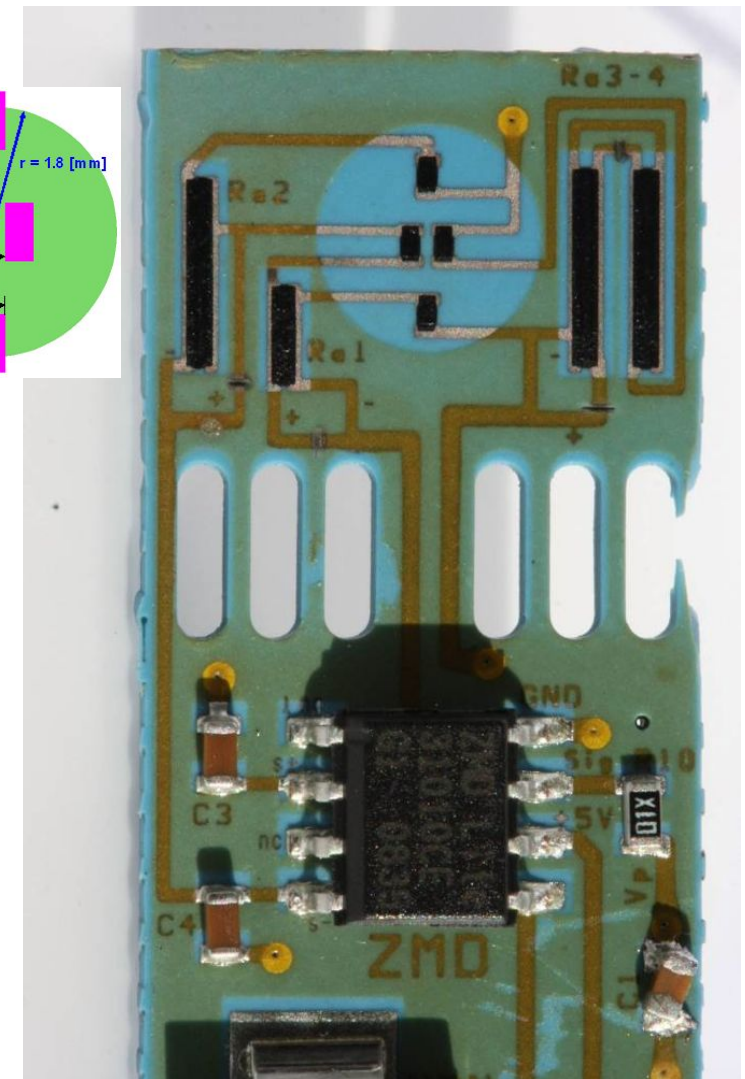
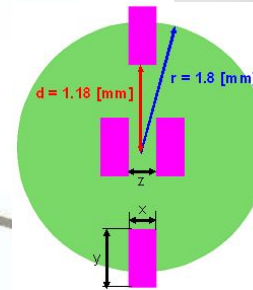
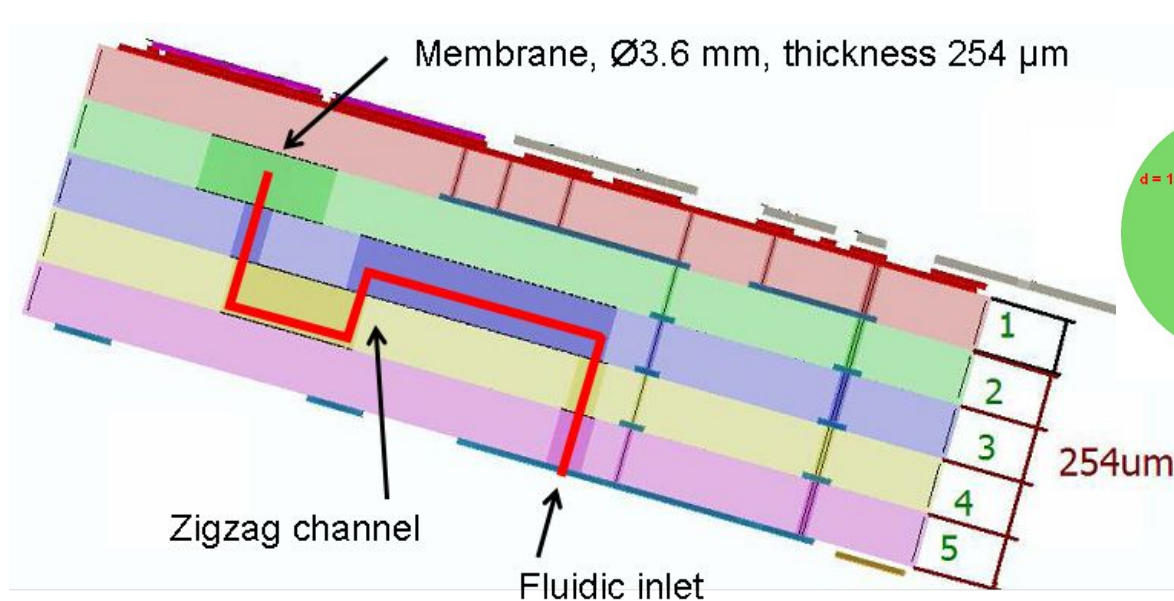
Membrane  
layout

- Nominal pressure: **6 bar**
- **Piezoresistive bridge** on circular membrane
- Pressure path to avoid stress concentration on membrane





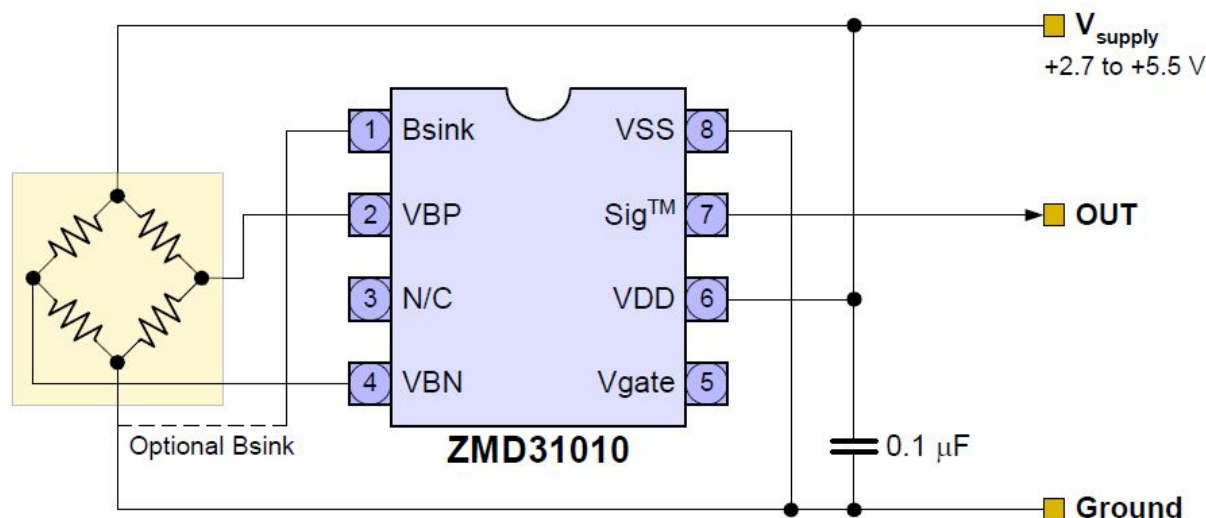
# Multisensor: pressure sensor



- Nominal pressure: **6 bar**
- **Piezoresistive bridge** on circular membrane
- Pressure path to avoid stress concentration on membrane

# Multisensor - pressure conditioning

- Easy adjustment of gain/offset
  - ⇒ Wheatstone bridge
  - ⇒ **programmable integrated conditioner** (*ZMD 31010*)

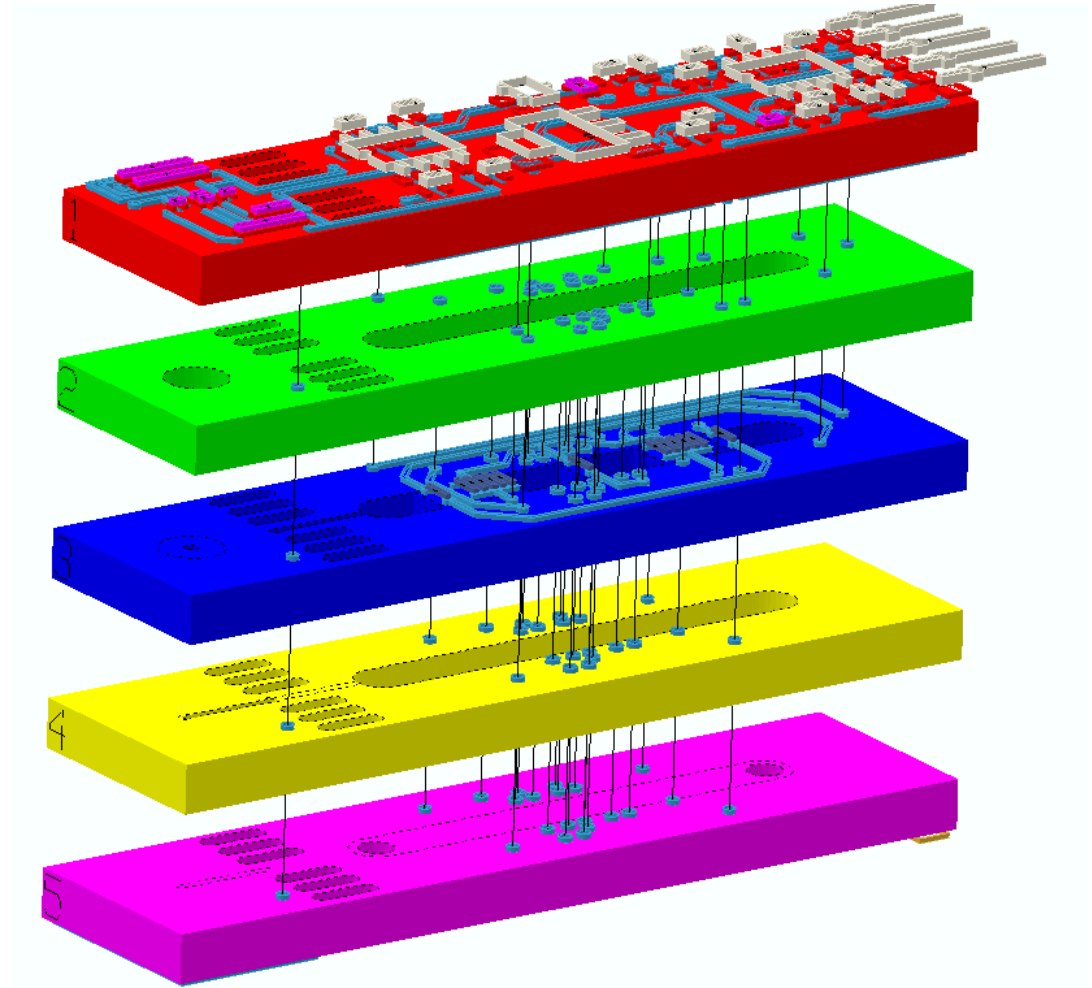




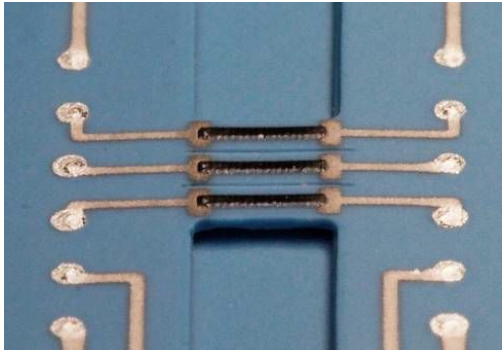
# Multisensor - LTCC stack

## LTCC DuPont 951

- **Vias**  
Ag *DuPont 6141*
- **Conductor tracks**  
Ag:Pd *DuPont 6146*  
Ag *DuPont 6145*
- **Resistors**  
10 k $\Omega$ /□ *DuPont 2041*  
PTC *DuPont 5092D*

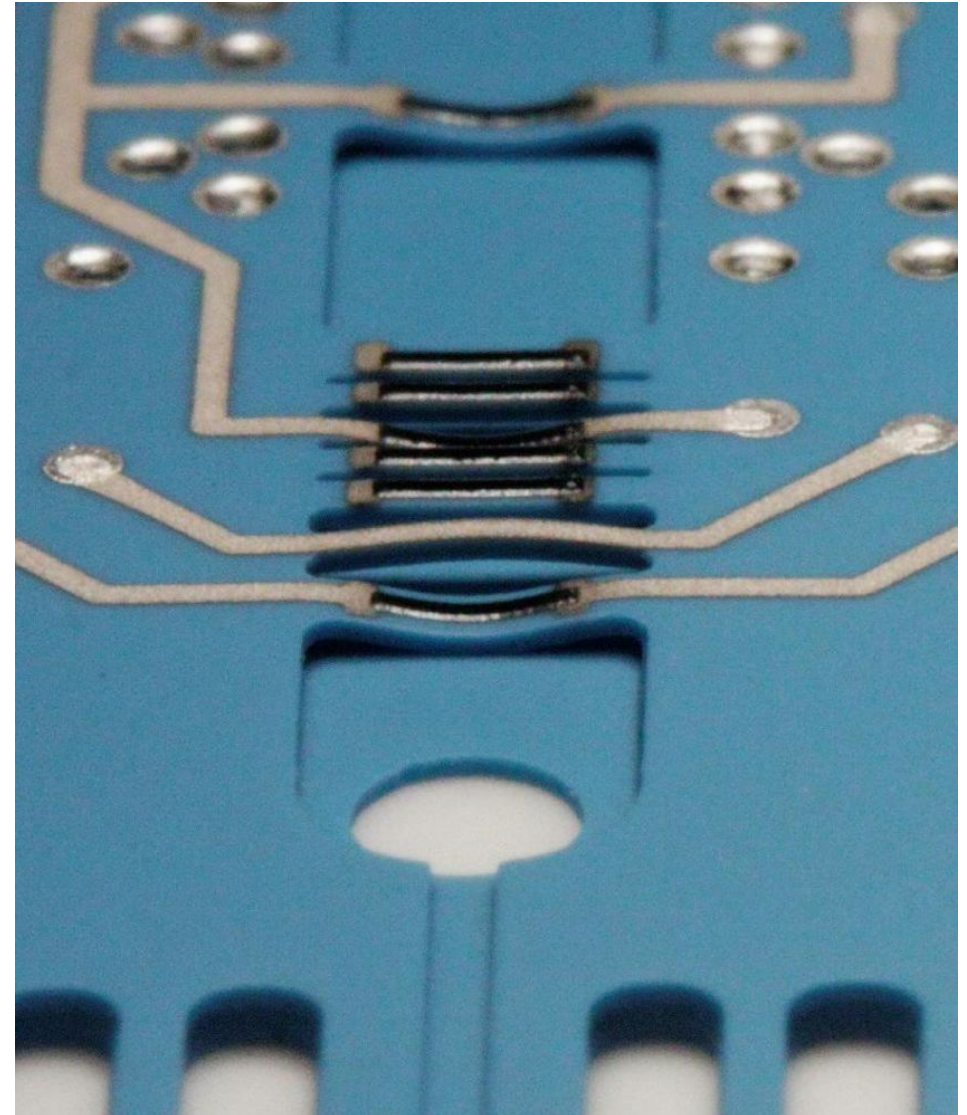


# Multisensor: manufacturing issues



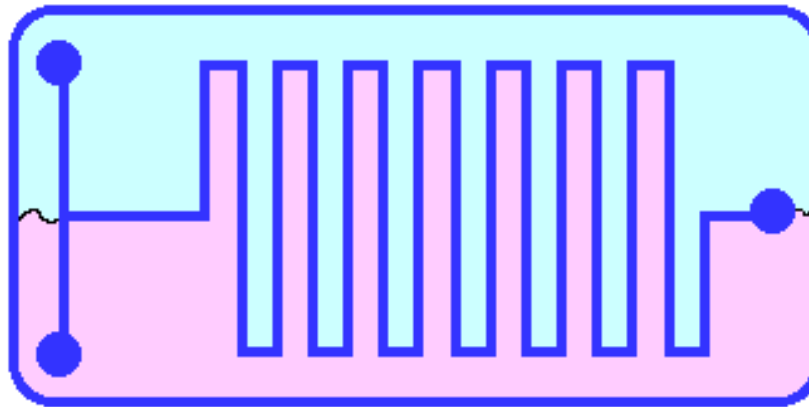
Differential sintering issues  
between LTCC and pastes  
→ **deformations**

⇒ Need adapted layout or  
sacrificial layers

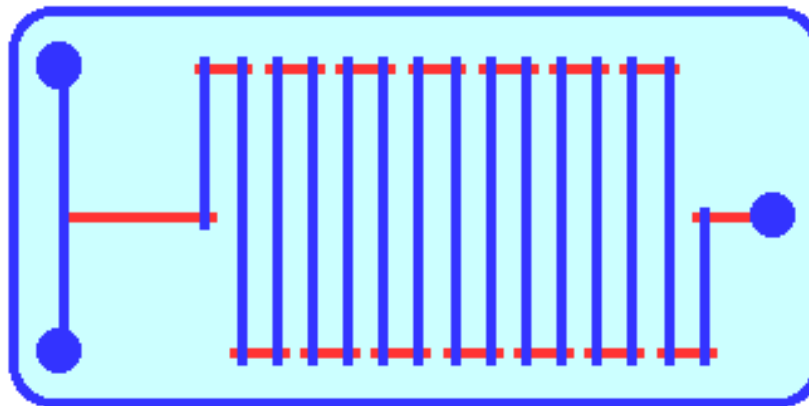


# Meanders & complex circuits

## Fabrication of complex LTCC fluidics



- LTCC sheet weak
- Strong risk of clogging



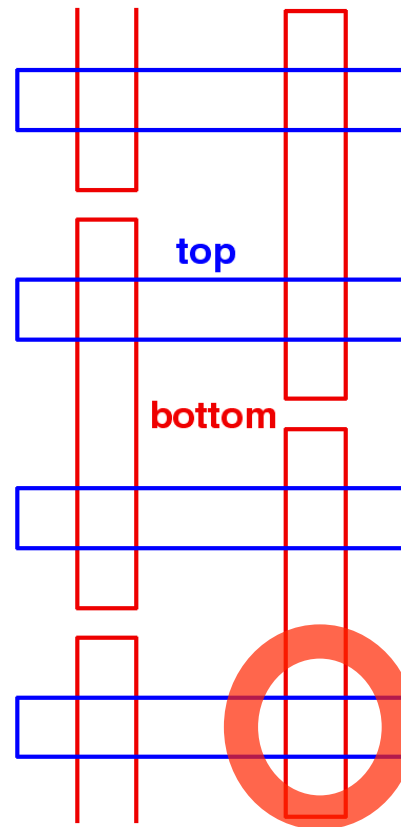
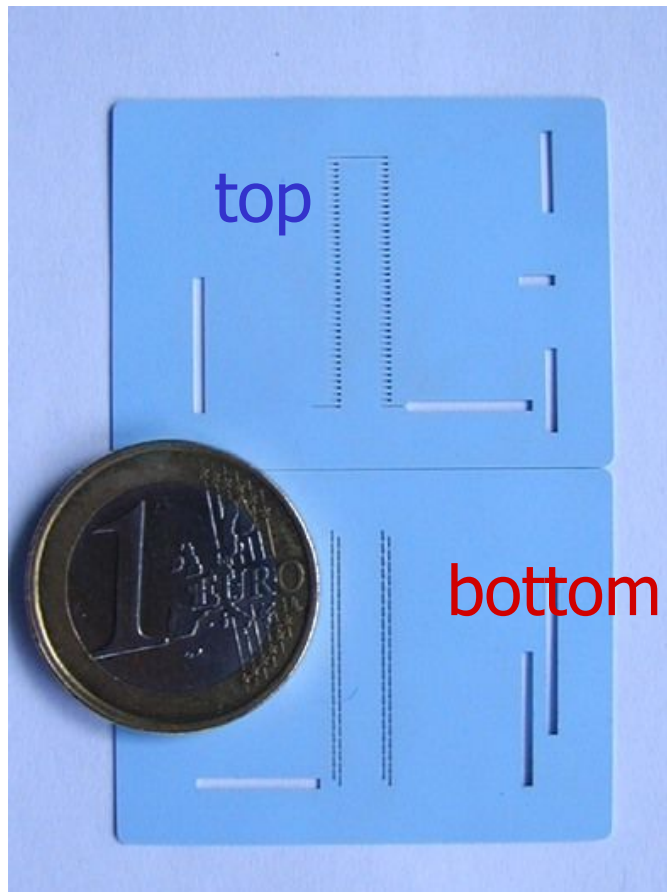
- LTCC sheet stronger
- Less risk of clogging

■ Sheet 1 (top)  
■ Sheet 2 (bottom)

⇒ Avoid long, narrow & windy cuts!

# Meanders: zig-zag micromixer

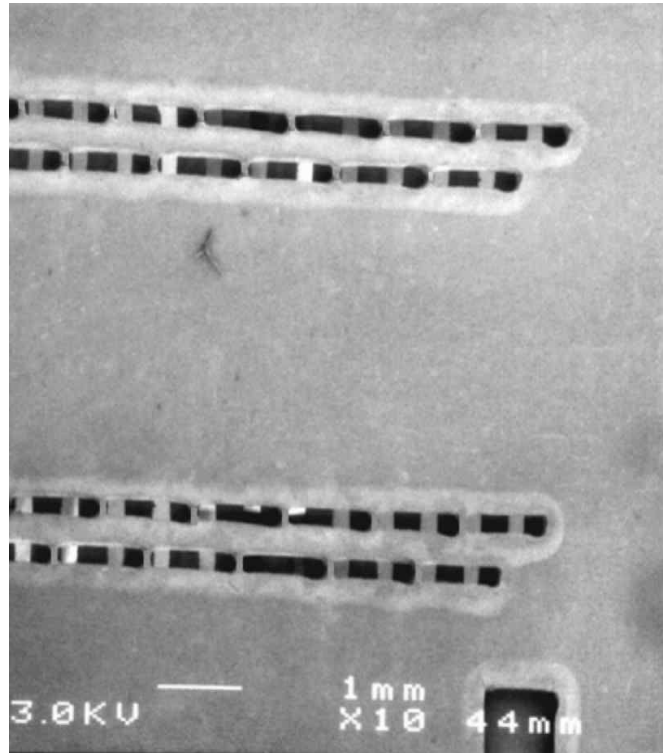
V. Mengeaud, EPFL 2002



- Two-layer “zig-zag” mixer
- Preserves integrity of LTCC layers
- Extra length: alignment tolerances

# Meanders: zig-zag micromixer

V. Mengeaud, EPFL 2002

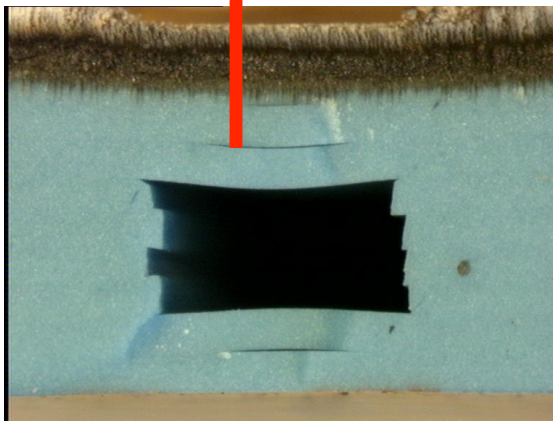
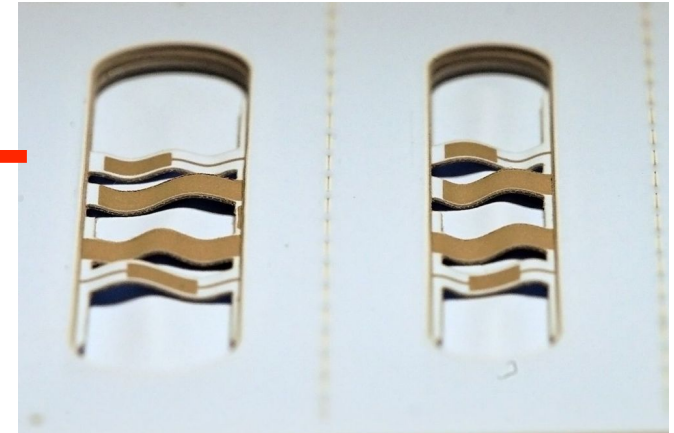


- Two-layer “zig-zag” mixer
- Preserves integrity of LTCC layers
- Extra length: alignment tolerances
- Reliable structure, no clogging
- + electrodes: electrochemical microreactor



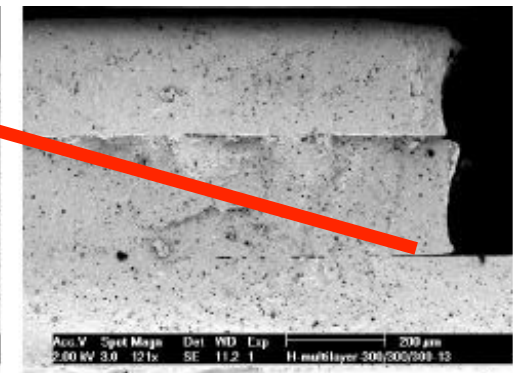
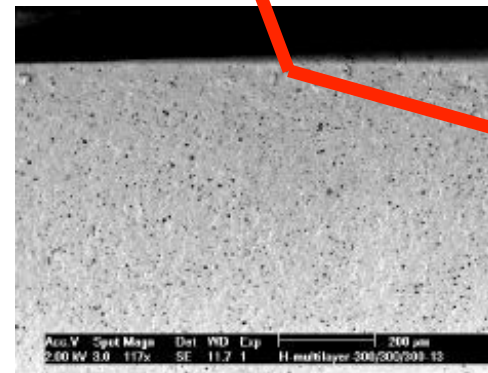
# Ceramic structuration: lamination issues

- Standard:  $\approx 70^{\circ}\text{C}$  20 MPa
- Deformation of intricate structures
- Lower pressure at edges
- Bonding above & below cavities



Multilayer  
above &  
below  
cavity

Bonding  
besides a  
cut-out zone



Basic problem: homogeneous LTCC behaviour  
Mechanisms of lamination  $\approx$  tape deformation  
Additional possible problems during firing

## 1. Introduction

## 2. Processing for 3D

## 3. Materials formulation

## 4. Limitations of LTCC

## 5. Conclusion & outlook

- **LTCC compatibility**
- **Carbon SVMs**
- **Mineral SVMs**
- **Adhesives for low-pressure lamination**

# Lamination: proposed solutions

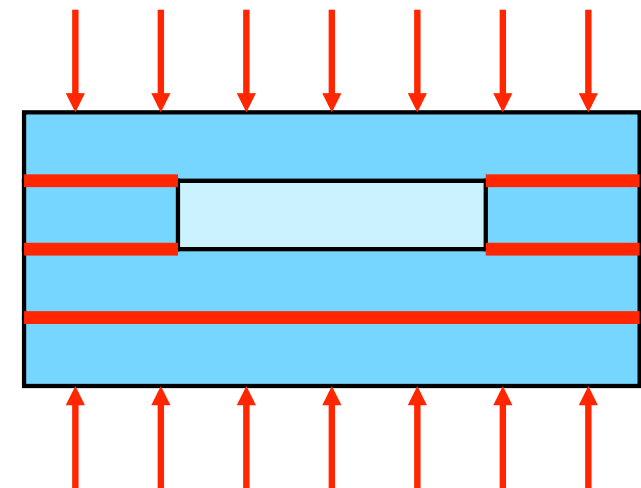
## Sacrificial volume materials - SVMs

- Prevent crushing / deformation
- Allow standard high-pressure/-temperature lamination
- Somewhat cumbersome (fill complex features)
- Ensure pressure transfer - good lamination



## Low-pressure lamination

- Careful determination of minimal parameters
- "Glues": solvents / thinners / honey / printing vehicles
- Adhesive tapes



# Formulation: solvents & LTCC

- **Classical - terpeneol, texanol**

- Excessive dissolution of tapes

- **Glycols - PG, BG, ...**

- Low attack
- Not suitable for PVB / EC
- Suitable for other binders

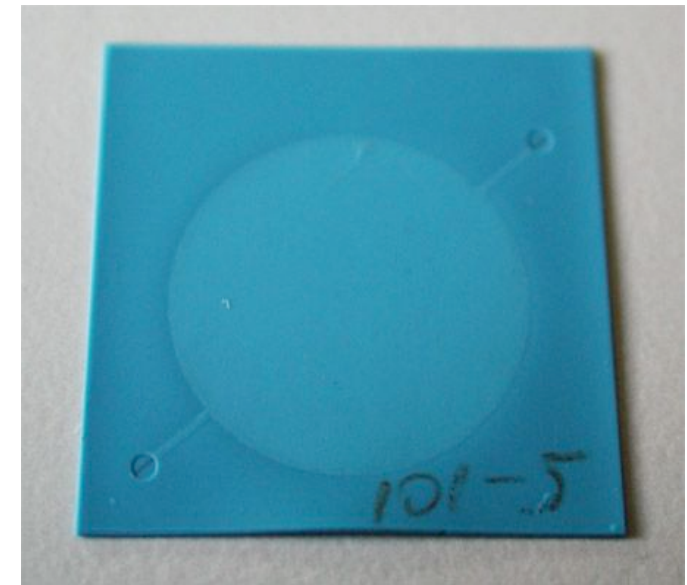
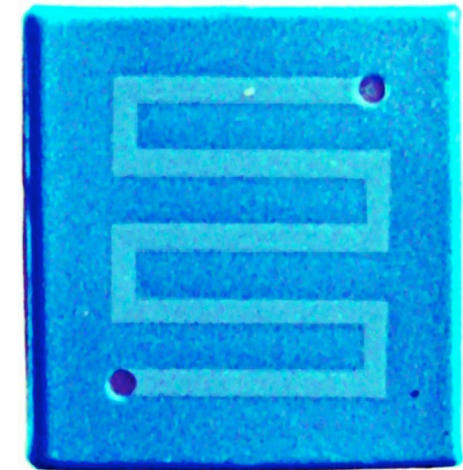
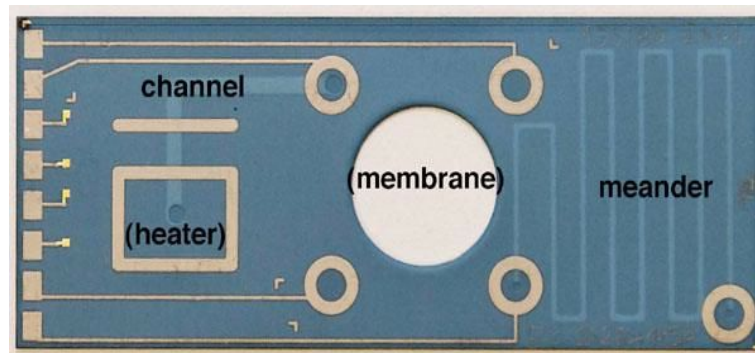
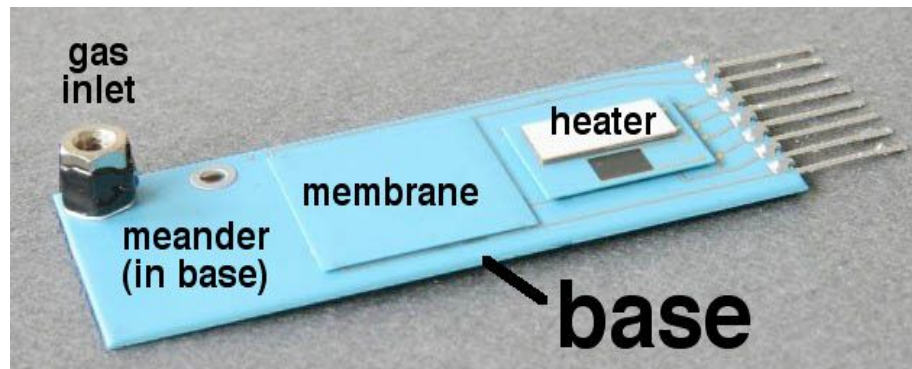
- **Fatty alcohols**

- (Cyclo)hexanol: good compromise
- + tailing solvent: lower volatility
- Suitable for PVB / EC (esp. with co-solvents / plasticiser)



# Carbon SVMs - applications

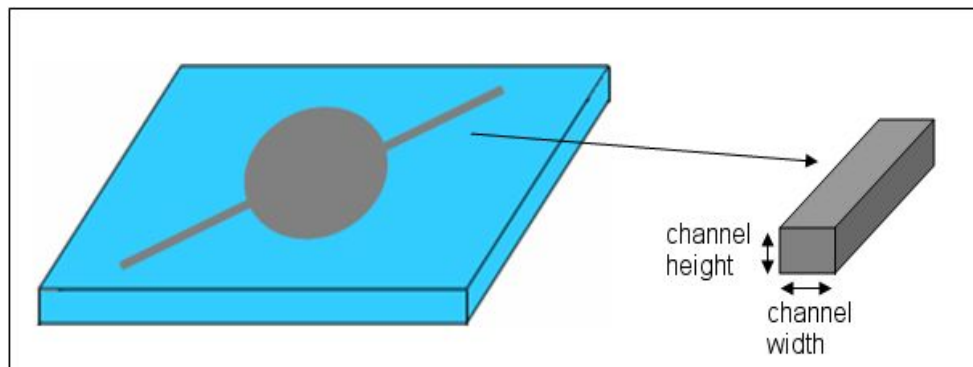
- Fluidic resistors
- Membranes
- Gas viscosity sensor
- Carbon: membrane & meander





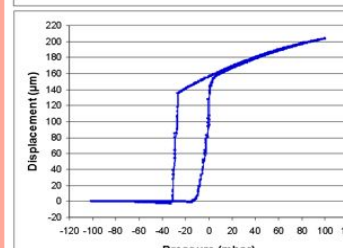
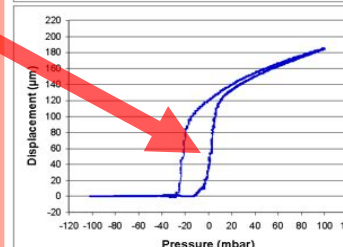
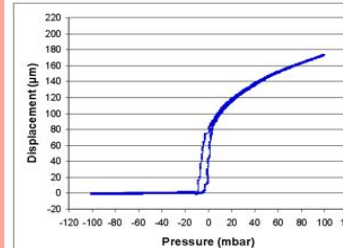
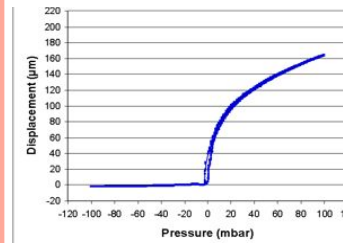
# Carbon SVMs - membrane issues

- LTCC membrane: 3x support + 1 thin ( $50\ \mu\text{m}$ ) DP 951 tapes
- Channel width (2:1) & height (2:1)  
→ Strong change of properties

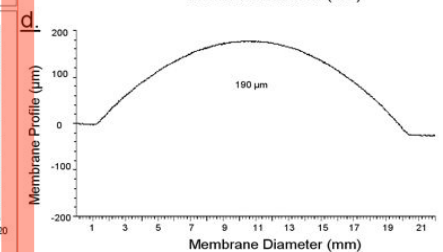
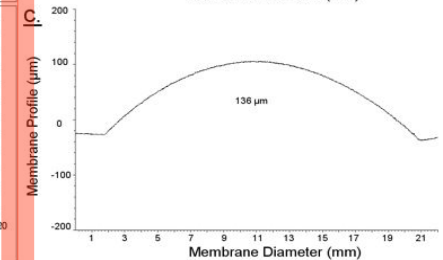
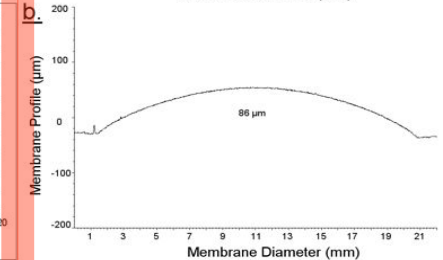
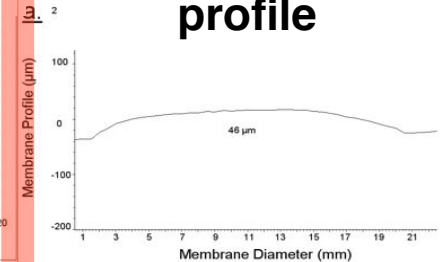


**Hysteresis**

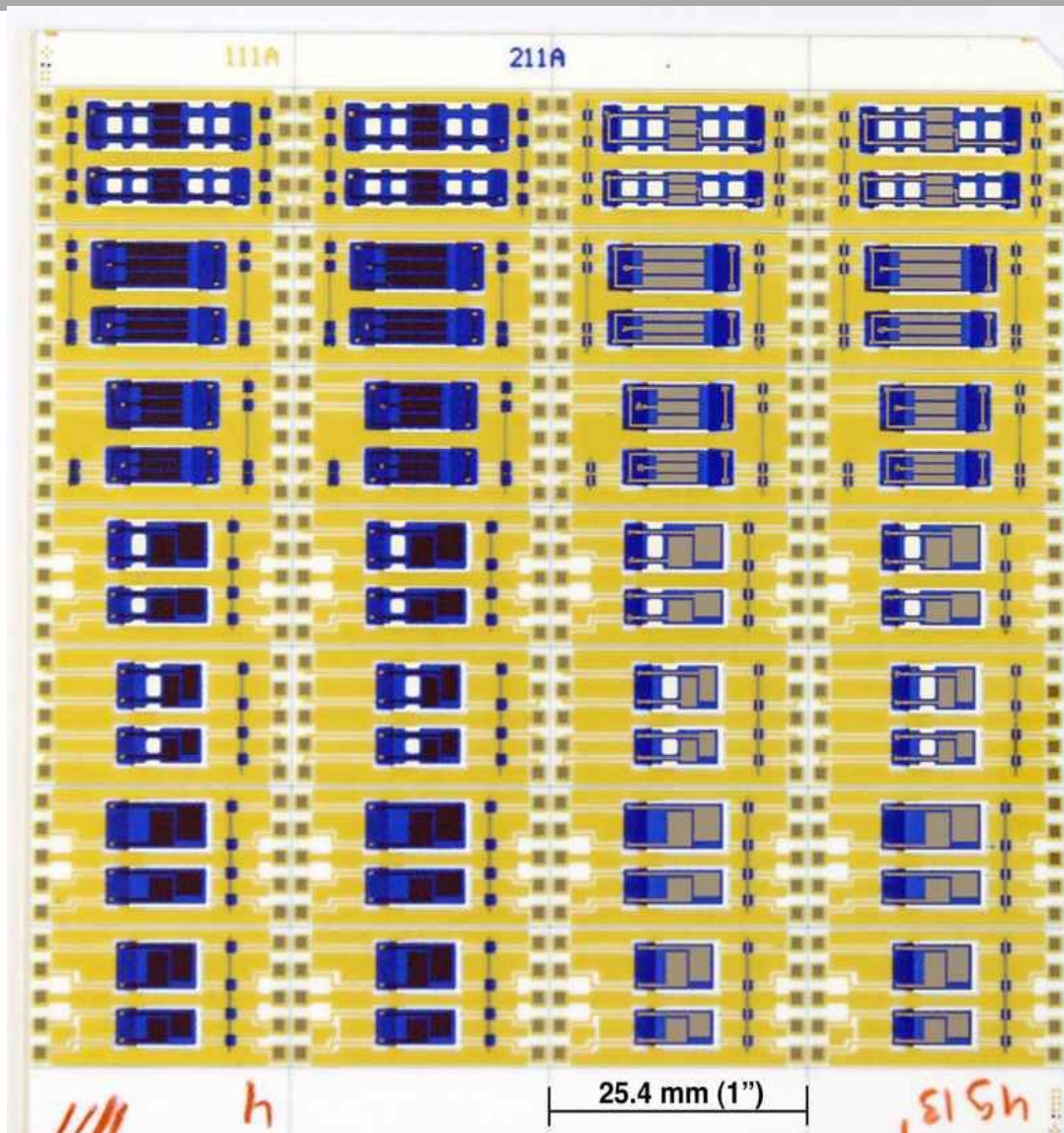
**Pressure-displacement**



**Membrane profile**



# Mineral SVMs – MgO-based

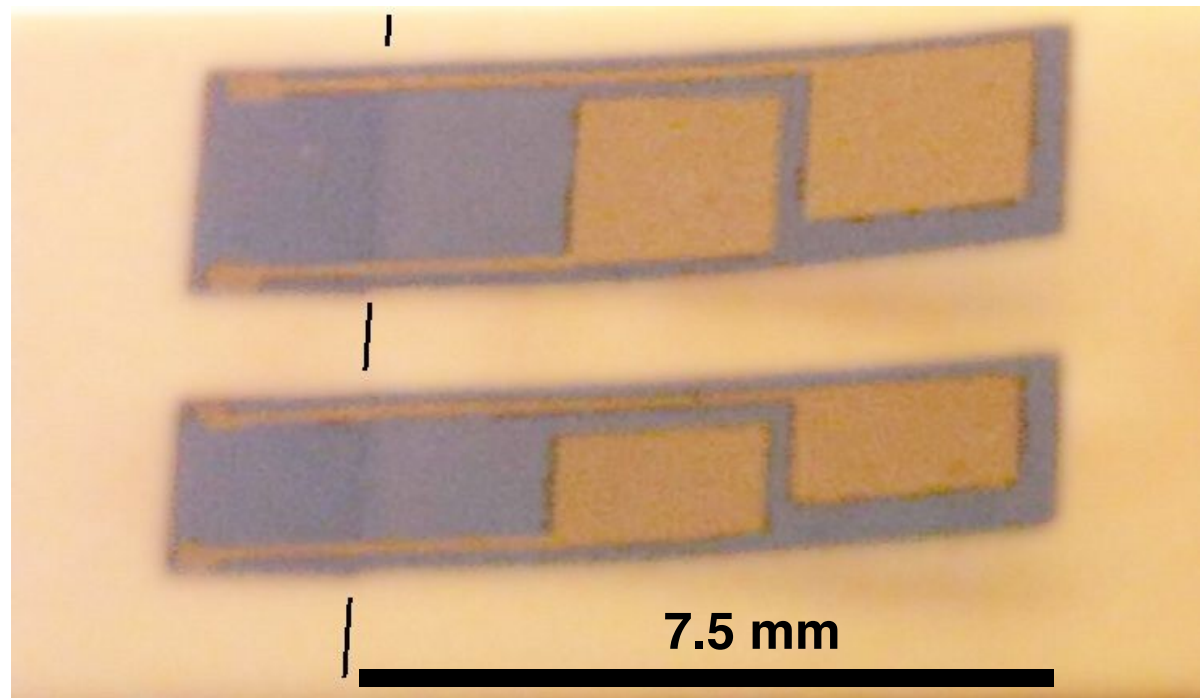


- Structures for capacitive force sensors / actuators
- Cantilevers or bridges
- Different width
- Plain or structured (cut-outs)

## Removal

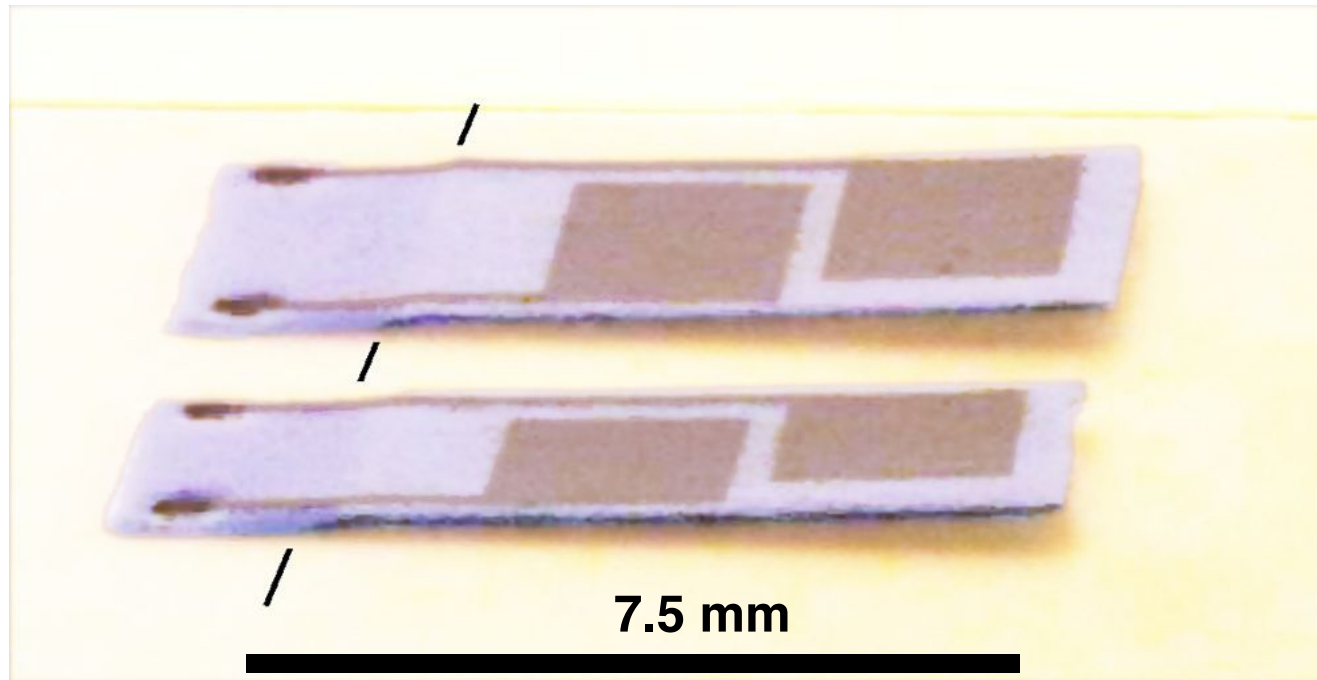
- Dip into acid ( $\approx 1$  day) @ RT
  - MgO not very reactive
  - "Dead-burned" or additives on surface
  - 10% phosphoric or acetic acid not optimised
- Rinse with tap water
- Neutralise acid with TRIS buffer
- Rinse with deionised water
- Rinse with isopropanol
- Dry @ 60°C in oven

# Mineral SVMs – obtained structures



- Plain cantilever : 2 x ESL 4913 + 1 x AgPd ESL 9635B
- Lifting due to built-in strain (TCE mismatch)
  - No lifting was present before etching

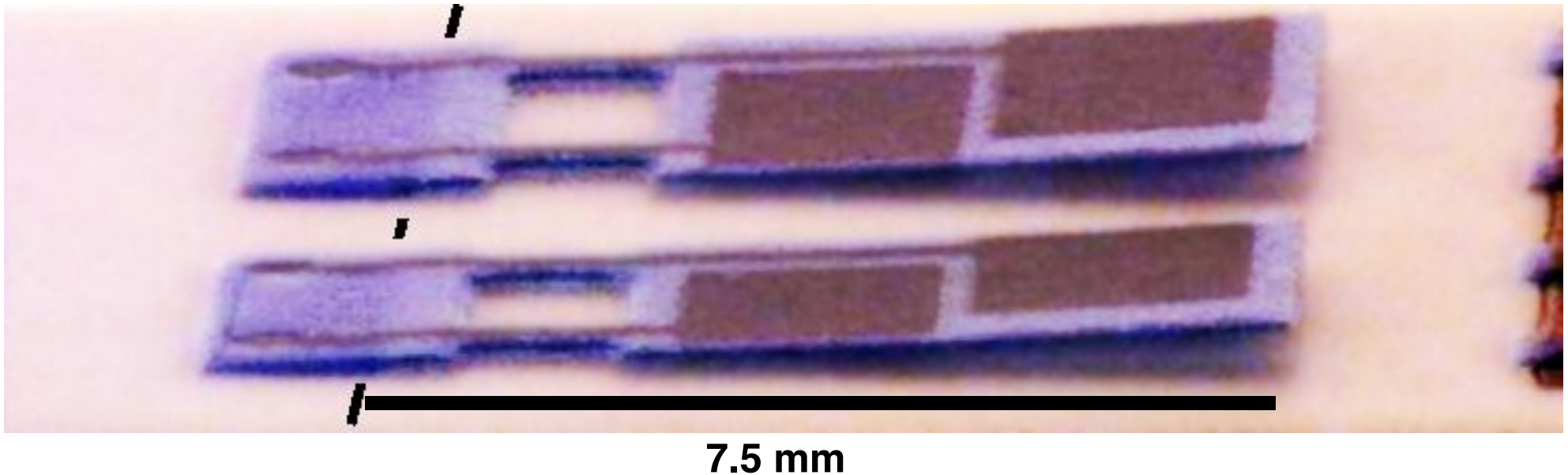
# Mineral SVMs – obtained structures



- Plain cantilever: 3 x ESL 4904 + 1 x 9535B + 1 x 4904
- Lower lifting due to partial structure compensation



# Mineral SVMs – obtained structures

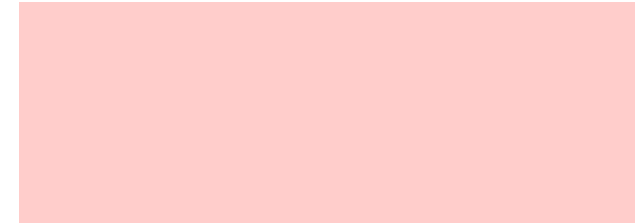


- Structured cantilever: "hinges" to increase flexibility
- Advantage: move weak point away from base-cantilever junction

## *2 alternatives:*

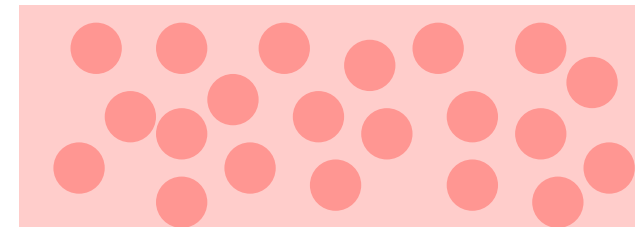
### ■ **Solvent-binder-plasticiser**

- Formulate to dry "just non-tacky"
- Upon reheating, plasticiser serves as solvent
- Somewhat progressive solid-liquid transformation (homogeneous glass)



### ■ **Solvent-binder-[plasticiser]-wax**

- Wax crystallises out @RT: non-tacky
- Upon reheating, wax acts as a solvent
- More abrupt solid-liquid transition possible



# Adhesives: binders

- "Common" binders selected
- PVB, PxMA or EC: used in tapes & pastes
- Hansen solubility parameters (give an idea...)
- Other binders possible (HPC, PVP, ...)

Binder	$\delta_d$ [MPa <sup>0.5</sup> ]	$\delta_p$ [MPa <sup>0.5</sup> ]	$\delta_h$ [MPa <sup>0.5</sup> ]	$R$ [MPa <sup>0.5</sup> ]	Note
PVB (20% OH)	18.6	4.4	13.0	10.6	Solubility in polar solvent with H bonding (alcohols, alcohol-ethers, alcohol-esters)
PMMA	18.6	10.5	7.5	8.6	<i>Solubility in esters, ketones, alcohol-ethers, alcohol-esters</i>
EC	17.1	7.3	9.7	9.0	Intermediate btw. PMMA & PVB In practice very easy to solubilise in a wide range of modestly polar solvents

# Lamination quality & distortion

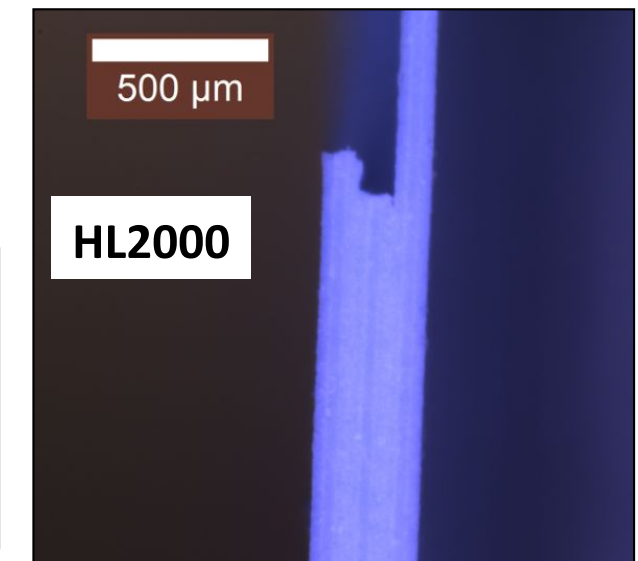
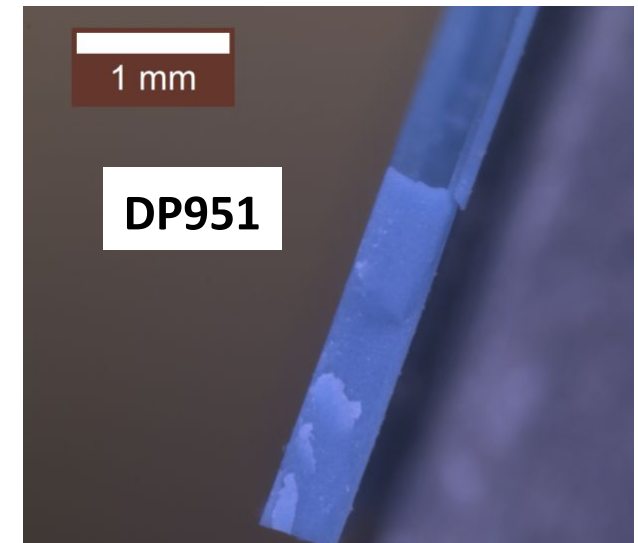
- Simple  $\varnothing 5$  mm membranes
- Tapes: DP 951 or Her HL2000
- Good bonding & low distortion @60°C

## DP951

Membrane ( $\mu\text{m}$ )		1.94 MPa		3.88 MPa		7.75 MPa	
Glue	LTCC	fired quality		fired quality		fired quality	
#344-5	1 <sup>st</sup> layer	7	dense	7	dense	4	dense
#346-6	1 <sup>st</sup> layer	12	dense	6	dense	10	dense
#362-4	1 <sup>st</sup> layer	6	dense	11	dense	7	dense
#363-2	1 <sup>st</sup> layer	11	dense	6	dense	3	dense

## HL2000

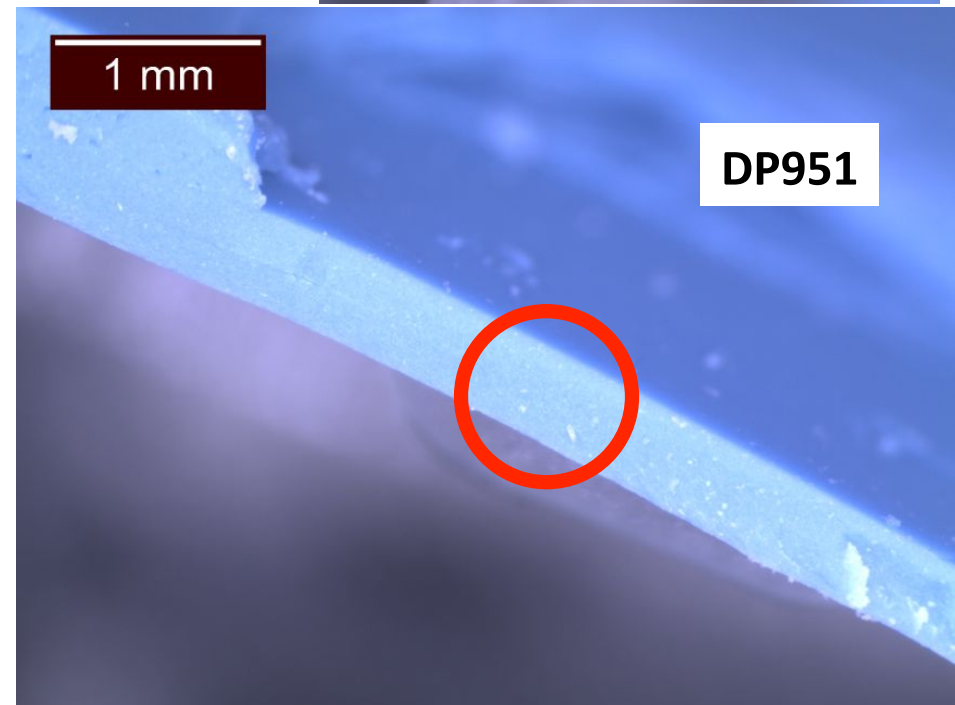
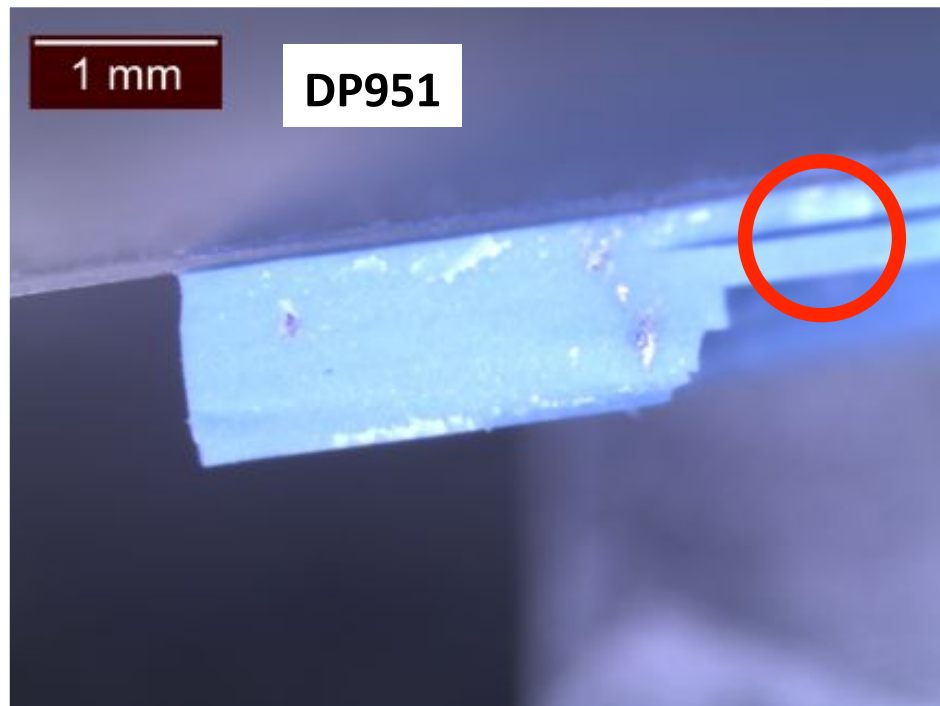
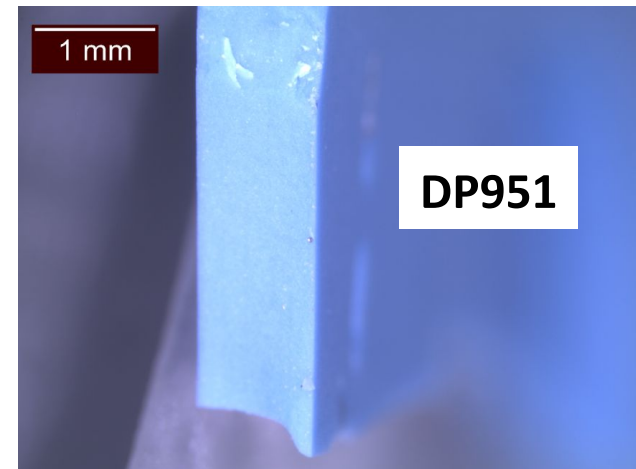
Profilometer results		1.94 MPa		3.88 MPa		7.75 MPa	
Glue	LTCC	fired quality		fired quality		fired quality	
#346-6	1 <sup>st</sup> layer	8	dense	10	dense	15	dense
#362-4	1 <sup>st</sup> layer	9	dense	5	little crack	13	dense
#363-2	1 <sup>st</sup> layer	10	little crack	13	dense	12	dense





# Lamination quality & distortion

- Body mostly OK
- Double membrane layer difficult (no pressure)
  - More glue: lamination OK, but deforms more upon firing



# Outline (4)

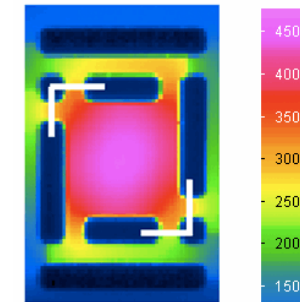
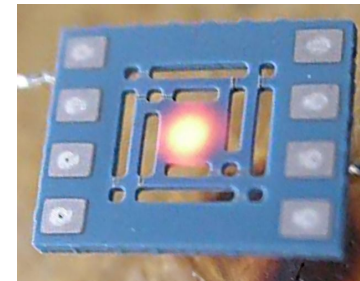
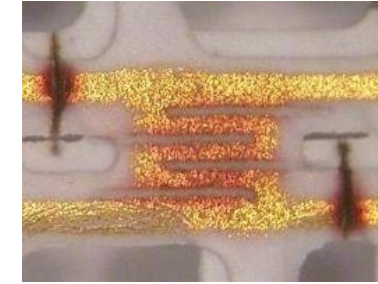
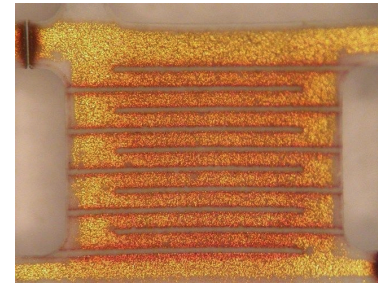
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1. Introduction
2. Processing for 3D
3. Materials formulation
- 4. Conclusion & outlook**

# Conclusions & outlook

## Thermal & fluidic structures

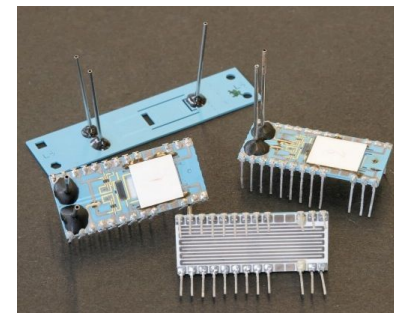
- Wide range of achieved structures, **BUT:**
- 3D structuration not trivial
- Techniques to be adapted to application
- Materials compatibility issues



LTCC  $\mu$ -hotplates  
*2010 Barras*

## Outlook

- More developments needed for better standardisation
- Long-term characterisation of reliability & stability



LTCC  $\mu$ -reactor  
*2005 Willigens*  
*2006 Maeder*

# Questions?



***Thank you for your attention!***

# Formulation: compatibility

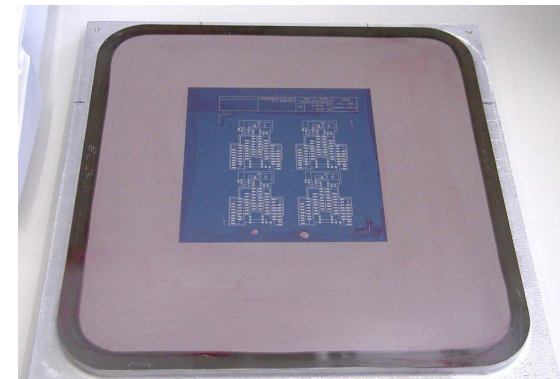
## ■ Tape binders (typ.):

- Acrylics: PMMA, PEMA, PBMA, ...
- Polyvinylbutyral (PVB)
  - > Sensitive to polar organic solvents



## ■ Screen emulsions (typ.):

- Polyvinylalcohol (PVA, PVOH)
  - > Sensitive to water only

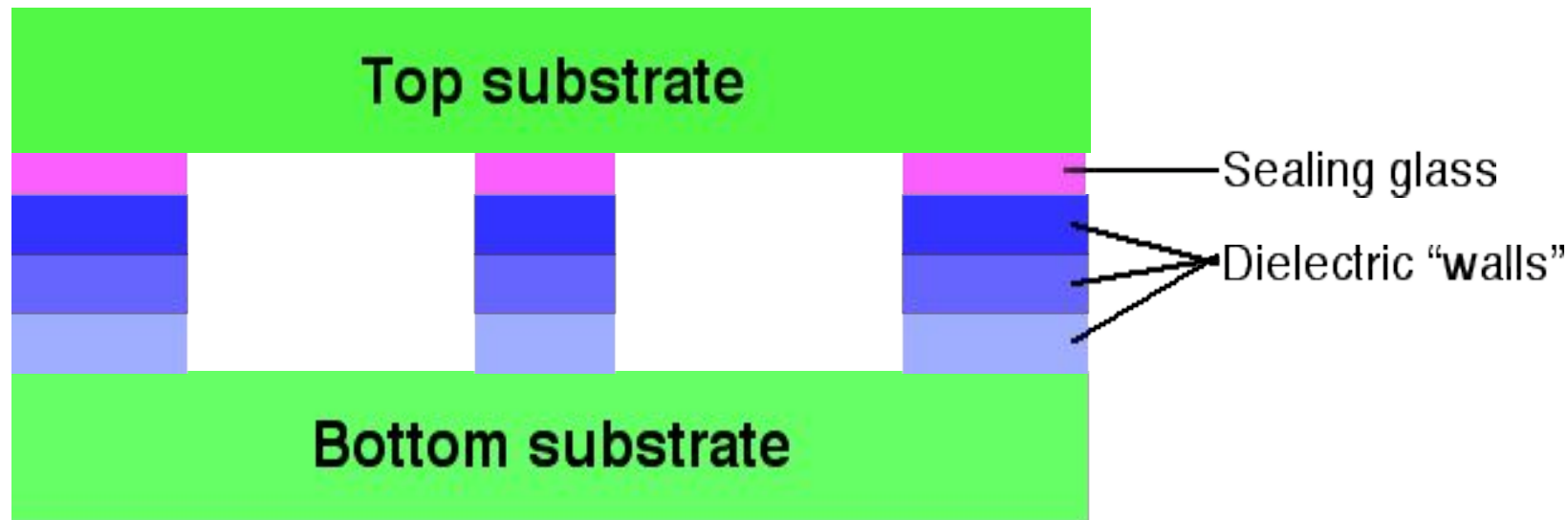


## ■ Avoid tape dissolution:

- Low solvent amount: high-solids
- Solubility parameter mismatch
- Balance volatility: screenability vs. persistence - attack
  - In this respect, methods compatible with volatile solvent better (spray, inkjet, dipping, ...)



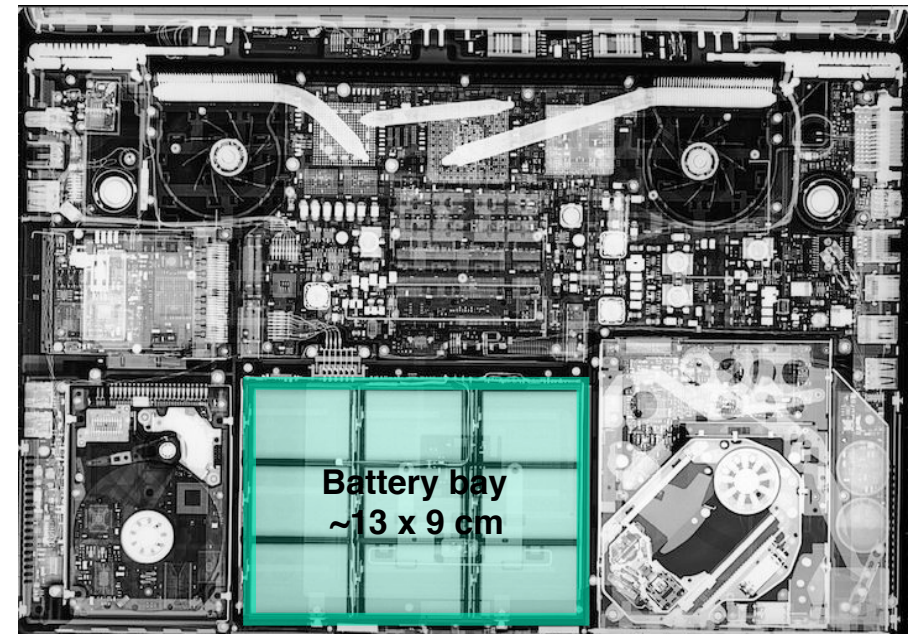
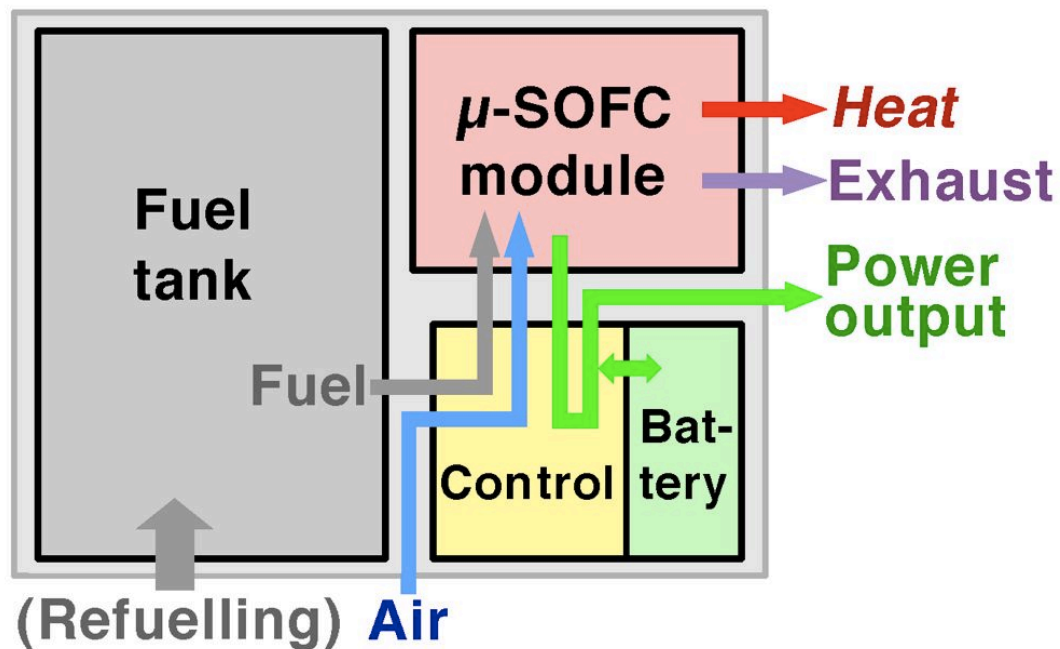
# Thick-film fluidics: glass sealing



- Alumina bottom substrate
- Walls built-up by thick-film dielectric
- Final layer = sealing glass
- Glass sealing of top substrate
- Max practical height: ca. 100  $\mu\text{m}$
- Simple circuits (1 layer)

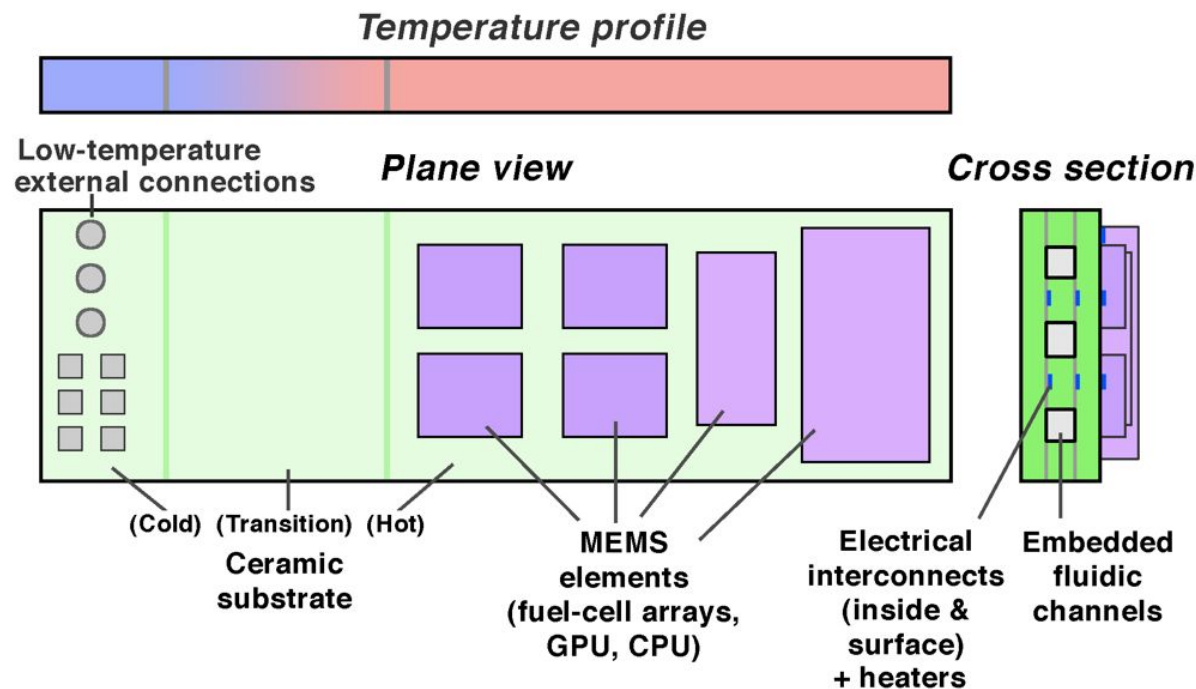
# $\mu$ -SOFC system principle

- Integration into a single, compact module
- Can replace battery bay in portable electronics



# LTCC $\mu$ -SOFC module concept

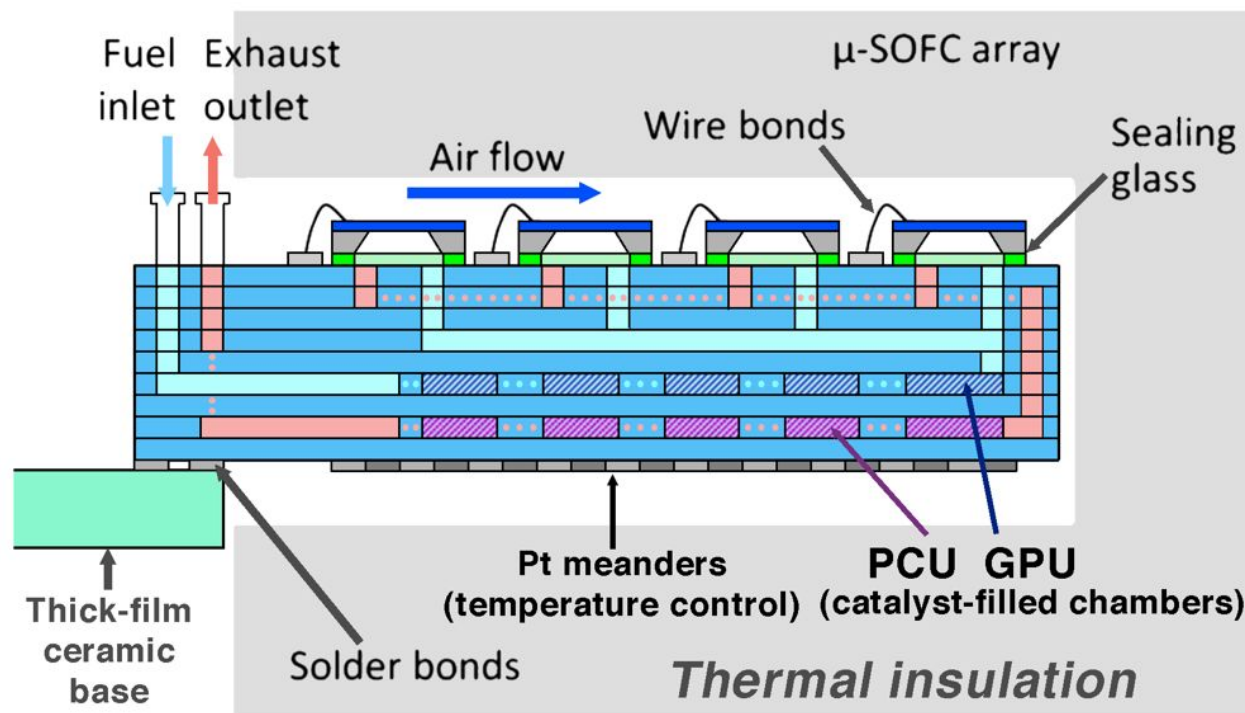
- Simple "stick" structure
- Facile thermal & mechanical decoupling
- External (electrical & fluidic) connections at low temperature
- Integration of fluidics & possibly gas processing



LTCC  $\mu$ -SOFC  
module packaging  
concept

# LTCC $\mu$ -SOFC module concept

- Simple "stick" structure
- Facile thermal & mechanical decoupling
- External (electrical & fluidic) connections at low temperature
- Integration of fluidics & possibly gas processing



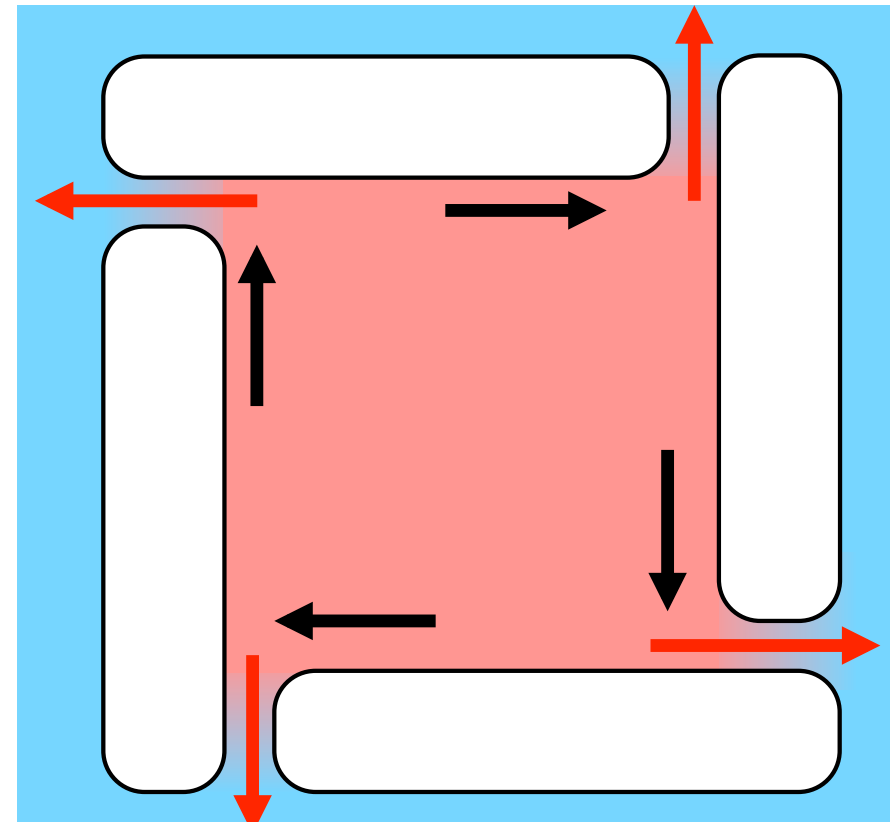
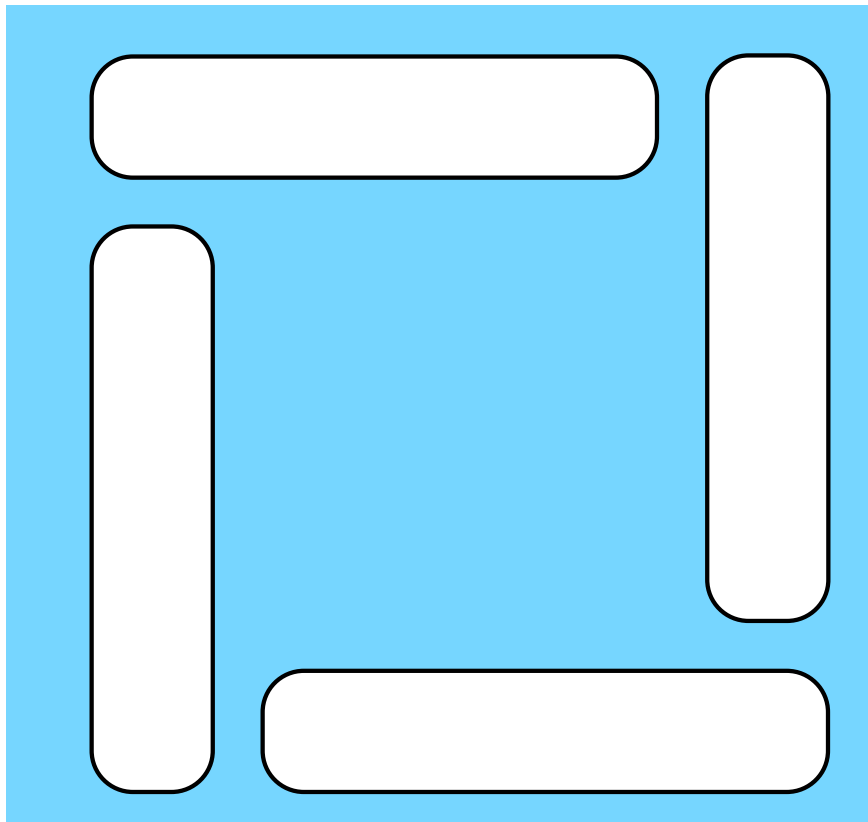
LTCC  $\mu$ -SOFC module

PCU = post-combustor

GPU = reformer

# Thermal decoupling

- Achieve thermal + mechanical decoupling
  - Free mechanical displacements ( $\rightarrow$ ) due to thermal expansion
  - Minimise thermal loss paths ( $\rightarrow$ )
  - Issues: processability - complete post-shaping not practical





# Carbon sacrificial layers - process

- Print [fire] sacrificial paste onto LTCC / ceramic substrate
- Laminate (LTCC) / print (ceramic) top layer
- Fire structure in air /  $O_2$ , OR:
- Fire structure in neutral gas & oxidise later
- Post-processing

